ENCOTEC QA
Revision 3.0
February 1993

QUALITY ASSURANCE MANUAL

Revision 3.0

Recipient ID: Michael Schmoldt - RMT, Inc.

Document Control ID: QA3072

Prepared by:

Environmental Control Technology Corporation

NOTICE

This document accurately presents the quality assurance philosophy and capabilities of ENCOTEC as of the date of publication. Revisions to any or all sections may occur at any time and at the sole discretion of ENCOTEC.

This manual, or any part thereof, is not intended for use without the expressed, written consent of ENCOTEC. Any liability associated with its unauthorized use rests solely with the user.

ENVIRONMENTAL CONTROL TECHNOLOGY CORPORATION (ENCOTEC)

QUALITY ASSURANCE MANUAL

Approved By:

Peter F. Atkins, Jr. - President — 3/1/93 Date

John E. Schenk - Vice President — 3/5/93 Date

Walter Roudebush - Technical Director — 3/5/93 Date

C. Michael O'Quinn - Laboratory Manager — 3-01-93 Date

Roger Roussel - Inorganic Chemistry Department Manager — 03/05/93 Date

James Kuehn - Organics Chemistry Department Manager — 3/05/93 Date

Stanley Smith - Quality Assurance Officer — 3/5/93 Date

Martha Frank - Technical Documentation Officer — 3/5/93 Date

Jane Hancock - Quality Assurance Chemist — 3/5/93 Date

TABLE OF CONTENTS

1.0 Introduction to ENCOTEC

Environmental Control Technology Corporation (ENCOTEC) is a wholly owned subsidiary of Rollins Environmental Services, Inc. (a Delaware corporation) and has provided comprehensive environmental consulting and analytical services for over twenty (20) years. ENCOTEC provides services in four functional areas: environmental chemistry, environmental engineering, risk assessment and pollution abatement. The firm has performed long-term, large scale investigations and surveys involving hazardous waste, hazardous waste sites and environmental monitoring for both public agencies and private sector clients. The distinguishing feature of our company is the capability to evaluate and define the scope of an environmental problem, determine the field monitoring program needed to document the situation, collect uncontaminated representative samples, perform the laboratory analyses, and then evaluate the data, assess the abatement, treatment or control options, and recommend cost effective solutions.

The firm's consulting activities extend into the areas of process recommendations and design, evaluation of existing facilities and processes, basic research, and technical and engineering services in the area of environmental quality and pollution abatement. Analytical services encompass essentially all parameters (physical, chemical, biological, and bacteriological) normally associated with hazardous waste characterization and environmental fate and effect. Specialized consulting services provided by ENCOTEC lie in the area of treatability studies, research and development projects, biological testing, mathematical modeling of environmental systems, and toxicology and risk assessments.

ENCOTEC currently houses its operations in two nearly adjacent facilities providing a total of 44,000 square feet. Both facilities are located in Ann Arbor, Michigan. The modern laboratory facilities, combined with state-of-the-art instrumentation and well-trained chemists, have enhanced ENCOTEC's Quality Assurance Program.

2.0 ENCOTEC Quality Assurance Policy Statement

2.1 Preface

At ENCOTEC, Quality Assurance/Quality Control (QA/QC) is an integral part of all services performed. The management of ENCOTEC has established and reinforces an attitude with regards to quality assurance that is consistent with the rigorous data quality objectives set for all samples analyzed. Although each project has unique data quality objectives, all projects have a baseline objective of technically defensible scientific data. It is the purpose of this document to define quality control measures that will be enforced for samples that are received by our laboratory.

The contents of the company's QA/QC protocols are the cumulative effort of many individuals and are the result of the company's years of experience and internal development. Thus, this document is to be considered as confidential information, and is not to be released, reproduced or distributed without prior authorization.

2.2 Philosophy and Policy Statement

ENCOTEC operates a multidisciplinary laboratory engaged in providing analytical and consulting services to a wide variety of industrial, private, local, state, and federal governmental agencies. Data generated and reported by the laboratory is used for many purposes such as engineering design of treatment processes, compliance with pollution control regulations, and toxicity and risk assessment. ENCOTEC's QA policies are built around the premise that all data must be scientifically valid and

able to withstand the rigors of litigation. With these goals in mind, ENCOTEC has developed a rigorous QA/QC program to assure that representative, comparable, complete, accurate, and precise measurements are made.

The personnel and management of ENCOTEC strongly maintain a philosophy consistent with the analytical services that they perform. This philosophy includes the following key elements:

a) A QA program will succeed only if each staff member understands and agrees upon the need for QA/QC. The quality assurance manual, therefore, is made available to all company personnel with at least one copy maintained within each department of the company.

b) A QA program is a written set of documents and procedures that must be followed to ensure data integrity. These guidelines are determined by consensus of the laboratory operations and technical management and reflect the ethical standards of the company. These documents are written to identify each employee's responsibilities in reaching the program's goals.

c) The purpose of a QA program is to monitor the entire data collection process. If a QC problem is pinpointed, then corrective action can be taken. The purpose of corrective action is to remedy a specific problem and prevent its recurrence. Corrective action can also serve as an educational tool for laboratory management as well as for specific personnel.

d) A QA program should not be static. The program should be in operation at all times and should be continuously evolving to meet the needs of data users.

2.3 Regulatory Mechanisms - QA/QC Driving Force

ENCOTEC believes that an appropriate QA/QC program promotes the development of and conformity to requirements that help ensure that "the work is done right the first time". These requirements define our approach to techniques that assure quality throughout all aspects of our work. "Doing it right" includes meeting or exceeding applicable ENCOTEC client or regulatory requirements and maintaining sufficient documentation to provide both scientific and legal defensibility. The enforcement of QA/QC guidelines provides for a grade of excellence in all data generated.

The federal government has taken the lead in producing a number of regulatory mechanisms aimed at monitoring, controlling and reducing pollution in our air, soil and water. While these pieces of legislation do not provide a baseline for an effective QA/QC program, they are the driving force behind most QA/QC policies. Therefore, QA/QC policies have been designed and implemented to meet or exceed all regulatory guidelines set forth by the federal government. In order to have a proper understanding of QA/QC policies one must understand the reasoning behind those policies. Summarized below are the major pieces of legislation that provide the driving force behind ENCOTEC's QA/QC policies.

2.3.1 CERCLA/SARA

The Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) as Amended in the Superfund Amendments and Reauthorization Act (SARA). These two acts, more commonly known as Superfund, are aimed at identifying and remediating contamination at NPL sites. Congress established the Superfund Program to deal with emergencies arising from abandoned wastes and waste sites, to provide long-term solutions to the most serious sites, and to encourage more responsible treatment of hazardous wastes in the future. Congress passed CERCLA in 1980 and SARA in 1986, with development in methodologies following soon thereafter. Most of the analytical work in response to this program is performed through the Contract Laboratory Program (CLP) or by using approved laboratories following the CLP Statement of Work (SOW) protocols.

2.3.2 RCRA

The Resource Conservation Recovery Act of 1976 (RCRA) was established to insure proper management of hazardous wastes. One of the major provisions of RCRA is to protect groundwater from hazardous material contamination (i.e. the migration of hazardous substances from waste sites into the groundwater). RCRA regulations also provide a framework for determining if a waste is hazardous, for treating a hazardous waste, and for determining the extent to which a particular site may be contaminated. The primary analytical methods required for RCRA monitoring are published in EPA Manual SW-846. This document delineates sample collection practices, sample preparation techniques and analytical procedures for solid wastes.

2.3.3 NPDES

The National Pollutant Discharge Elimination System (NPDES) was established by the Federal Water Pollution Control Act Amendments of 1972 (commonly referred to as the Clean Water Act). NPDES is the primary regulatory mechanism for controlling wastewater discharges into U.S. waters. Any facility discharging process waters into U.S. waters must obtain a permit and monitor its effluent for specified pollutants. The methods of analysis for these effluents are outlined in 40CFR Part 136.

2.3.4 SDWA

The Safe Drinking Water Act (SDWA) of 1974 and its subsequent amendments in 1977 and 1986 was established to insure the provision of safe drinking water to the public. Primary and secondary drinking water standards were established and specific Maximum Contaminant Levels (MCLs) were set for a wide and expanding range of substances. In addition, the SDWA provided that operators of public water systems would monitor the quality of the water and treat it, if necessary, to assure that the concentration of any comtaminant is below the MCL established by the EPA.

2.3.5 OSHA

The Occupational Safety and Health Act of 1970 created the Occupational Safety and Health Administration (OSHA). Soon thereafter OSHA promulgated Permissible Exposure Limits (PELs) for a wide variety of chemicals present in the workplace. These PELs were adapted from the 1968 Threshold Limit Values (TLVs) of the American Conference of Governmental Industrial Hygienists (ACGIH). In addition, some consensus standards from the American Standards Association (now the American National Standards Institute - ANSI) were also adopted at the same time. While methodologies and QA practices are not as well defined as in other regulatory mechanisms, a variety of standard methods are employed by many laboratories. These methods include the NIOSH Manual of Analytical Methods, ASTM and EPA 600 series supplement Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.

List of Acronyms

ACGIH	American Conference of Governmental Industrial Hygienists
ANSI	American National Standards Institute
APHA	American Public Health Association
ASTM	American Society for Testing Materials
AWWA	American Water Works Association
BNA	Bureau of National Affairs
BTU	British Thermal Units
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	Code of Federal Regulations
CLP	U.S. EPA Contract Laboratory Program
DOT	Department of Transportation
HSWA	Hazardous and Solid Waste Amendments to RCRA
Landban	Land Disposal Restrictions
NIST	National Institute of Standards and Technology (formerly NBS)
MCL	Maximum Contaminant Level
NIOSH	National Institute For Occupation Safety Health
NPDES	National Pollutant Discharge Elimination System
OCL	Organochlorine
OP	Organophosphorus
OSHA	Occupational Safety and Health Administration/Act
PCB	Polychlorinated Biphenyls (as Aroclors)
PEL	Permissible Exposure Limit
QA	Quality Assurance
QC	Quality Control
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act

SARA	Superfund Amendments and Reauthorization Act
SOP	Standard Operating Procedure
SOW	Statement of Work
Superfund	CERCLA or SARA
SWDA	Safe Water Drinking Act
THM	Trihalomethane
USEPA	United States Environmental Protection Agency
WPCF	Water Pollution Control Federation

3.0 Quality Assurance - Goals and Objectives

3.1 Introduction

The scope of this QA/QC Program generally encompasses the policies issued in the 1979 edition of the USEPA/EMSL Handbook for Analytical Quality Control in Water and Wastewater Laboratories, and quality control guidelines as provided in the USEPA SW-846 Test Methods for Evaluating Solid Waste, third edition. Often, however, the QC program may be made more rigorous depending upon the specifications of particular contracts.

The Chemistry Section QA/QC Program incorporates two primary elements as follows:

1. The control of measurements: the continuous assurance that measurements and data values from the laboratory are generated when the analytical system, whether it is wet chemical, instrumental, or gravimetric, is operating within control. To do this, precision and accuracy limits for analytical methods are developed and tracked.

2. The control of laboratory operations: the continuous assurance that all analytical systems (wet chemical, instrumental, or gravimetric) are high quality, and well-maintained operations from start to finish.

Clearly, the key to implementing a QA/QC Program is in providing good management of these integral operations. The management at ENCOTEC has demonstrated a firm commitment to implementing this program by specifically budgeting manpower and

money in support of a broad-based quality assurance effort. Specific procedures for QC are written for each of the various methods used by ENCOTEC. In addition, all projects have project managers appointed for the project.

The goal of this QA policy and outline is to provide a general framework for all laboratory procedures performed by ENCOTEC. Specific methods and quality control are applied on a project-by-project basis depending on the client's needs. Specific standard operating procedures (SOP) are companion documents to this policy. Details of each method's QC requirements are provided in the SOPs. In addition to laboratory SOPs, project-specific quality control plans and SOPs are developed when required for programs such as the USEPA CLP projects, RCRA facility investigations, and similar projects. This policy statement provides the outline and general content for the development of these companion documents.

3.2 Quality Assurance Objectives - Laboratory Analysis

The analytical procedures performed by ENCOTEC include the analysis of environmental samples for organic and inorganic compounds. The quality assurance objectives are to produce data of known accuracy, precision, representativeness, and overall comparability which will complement the sampling and evaluation portions of any project. The goal of the QA program is to produce defensible data which meets or exceeds the USEPA program guidelines such as the Superfund Program under CERCLA, RCRA investigations using SW-846 methods and NPDES monitoring. In order to accomplish these goals, USEPA methods and quality control limits are used by the laboratory. Copies of applicable methods (based on USEPA Methods) and QA requirements are available in the laboratory. In

addition, ENCOTEC's general QA program and custody protocol will be followed to ensure sound laboratory operation. Any deviations from existing procedures are approved through the Technical Director.

The frequency of quality control effort with respect to blanks, spikes, and duplicates is summarized in Table 3.1. Quality control limits are also specified in most analytical methods. These control limits should be met. However, if any results are out of window, the results will be thoroughly reviewed, corrective action taken where necessary and samples reanalyzed where appropriate. Comparability of data for laboratory results is ensured by reporting all data in standard units.

Data completeness is insured by providing a final data sheet(s) for each sample result and a copy of appropriate QC results. All data is reviewed by a senior chemist and by the project manager to ensure that all requested QC data is present and all checks meet the established criteria. Finally, all appropriate QC data, if requested, is displayed in tabular form with QC limits in the report for client review.

The following table is a general summary of quality control frequency. A more complete definition of the QC frequencies plus additional QC specific to each department will be found in the analytical method SOPs. An analytical batch consists of a maximum of 20 samples or a month's accumulation of samples, whichever is more frequent.

Table 3.1
Quality Control Minimum Frequency Summary

Type	Description	Inorganics	Organics
Blank	Method or preparation	One per preparatory/ analytical batch	One per preparatory/ analytical batch
Duplicate	Duplicate of sample	One per analytical batch	-------
Laboratory Control Sample	Analyte-fortified blank	One per analytical batch	One per analytical batch
Laboratory Control Sample Duplicate (if requested)	Analyte-fortified blank	One per analytical batch	One per analytical batch
Matrix Spike	Analyte-fortified sample	One per analytical batch	One per analytical batch
Matrix Spike Duplicate	Duplicate of analyte-fortified sample	--------	One per analytical batch

4.0 QA/QC Organization and Responsibility

4.1 Introduction

Inherent in any successful quality assurance program is a system whereby the laboratory maintains the level of quality control necessary to render analytical results scientifically defensible. In addition, the coordination of efforts within the organization to ensure the validity of such information is a dynamic rather than a static process. Quality assurance and control must play an everyday role in every department within the organization.

Examination of the processes involved both through internal audits and external audits conducted by the USEPA, state agencies, and/or clients serve to strengthen the quality assurance program. An audit is an additional feedback mechanism as well as an examination of current procedures. This feedback motivates the organization to modify or improve existing practices and/or to implement new ones. It is in this way that ENCOTEC's quality assurance program can respond to increasingly sophisticated analytical requirements, to the rapidly growing environmental industry, and to its clients.

Quality assurance is central not only to the proper functioning of ENCOTEC's analytical laboratories but to every department. The following discussion outlines a summary of each department's role and interdepartmental interactions that help to ensure the integrity of data generated on behalf of our clients.

4.1.1 Chemistry Administration

- Provides guidance (managerial and technical) to other departments so that they may fulfill their QA/QC responsibilities.
- Coordinates and approves capital equipment expenditures and facility improvements to respond to analytical needs.
- Approves Quality Assurance Proposal Plans (QAPP) which address special client needs.
- Balances client needs with individual department staff and technical capacities in order to minimize work overloads which may have an adverse effect on data quality.

4.1.2 Project Management

- Communicates with clients to ensure that their needs are compatible with laboratory capabilities.
- Communicates with laboratories to facilitate the timely scheduling of analytical work, thereby reducing the occurrence of work overloads which, in turn, affects data quality.
- Compiles analytical data from each laboratory, provides final review of data for reasonableness, for overall accuracy, and transmits finalized data to the client.

4.1.3 Technical Management

- Provides the primary oversight for the QA/QC program.
- Communicates with the analytical laboratories as well as the laboratory management regarding the proper implementation of QC and analytical methods.
- Provides primary oversight for methods development and research as well as project-specific research.

4.1.4 Analytical Laboratories

- Implements all testing requests.
- Implements all QC procedures.
- Ensures that projects are completed within the pre-defined time frame.
- Ensures that analyses are completed within the QC guidelines presented in the analytical methodologies.

4.1.5 Management Information Services Group (MIS)

- Responsible for implementation and ongoing development of the Laboratory Information Management System (LIMS).
- Provides software and computer systems support to all departments. All departments are dependent upon accurate and rapid communications and information transfer.
- Furnishes the proper computerized archiving of summarized data, quality control information, standard operating procedures (SOP), and other supporting documentation.
- Ensures that archived data and quality control information is secure.

4.1.6 Laboratory Support Group (LSG)

- Provides the proper preservation/storage, receipt, and disposal of samples received for analysis by the laboratory.
- Initiates a chain of custody for every sample received or validates an existing one.
- Works with project managers in assigning the correct analytical procedures to each sample and in communicating this information to all appropriate laboratories.

4.1.7 Environmental Engineering and Assessment (EE&A)

- Provides engineering consulting services enabling clients to arrive at the most technically sound decision regarding their environmental concerns.
- Obtains environmental samples under contract to clients for analysis by ENCOTEC.
- Works closely with project management in evaluating analytical data submitted by laboratories and provides the necessary feedback to the laboratories as to the quality of that correlated data.

4.1.8 Clerical

- Provides report transcription capabilities, converting handwritten data to computer-generated reports.
- Provides an additional check on completed data with regard to proper data format and structure.
- Transmits finalized data to clients following review by project management and is a critical link in the data transfer process.

The overall Quality Assurance Program relies heavily upon the interrelationships between the various functional groups within the company. Effective communication which is integral to the entire QA effort is not a simple or linear process, but involves a series of communication mechanisms that address the responsibilities and needs of each functional group. Within every QA directive, communication and feedback are of paramount importance. Due to the nature of this process, the operation is dynamic and is continuously being improved to meet the needs of the company.

4.2 Quality Assurance Management Structure

Management organization at the Company, Chemistry Section, and Laboratory levels are illustrated in Figures 4.1, 4.2, 4.3, and 4.4, respectively. Paralleling the Chemistry Section management structure, ENCOTEC's Quality Assurance Organization (Fig. 4.5) is coordinated by the Technical Department. Continuing development of the company's QA program occurs through the Technical Director and Quality Assurance Officer. Implementation involves laboratory operations and, to a lesser extent, project management. Communication and feedback are frequent and at many levels.

ENCOTEC
Management Organization

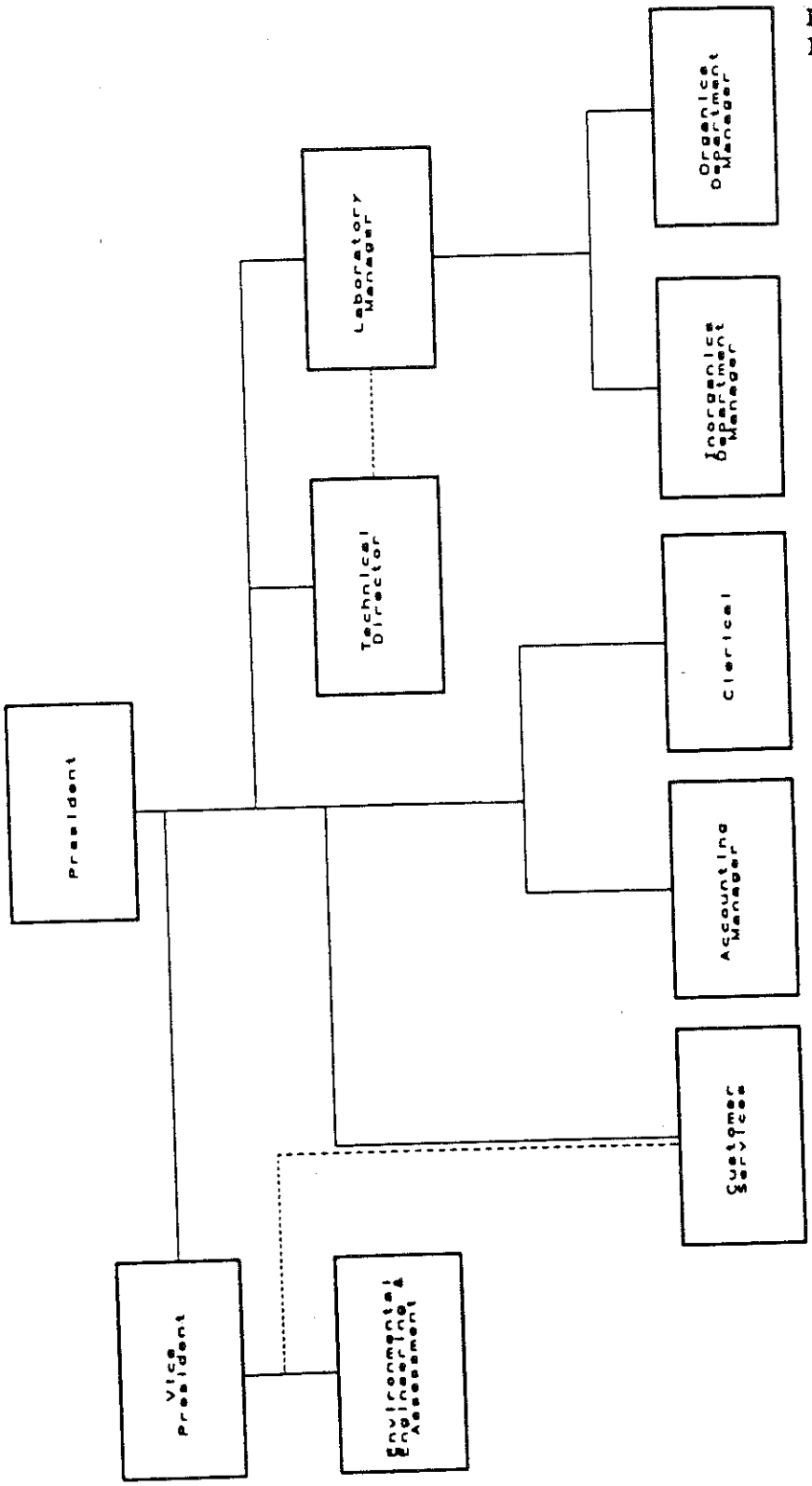


Fig. 4.1

Chemistry Administration Organization

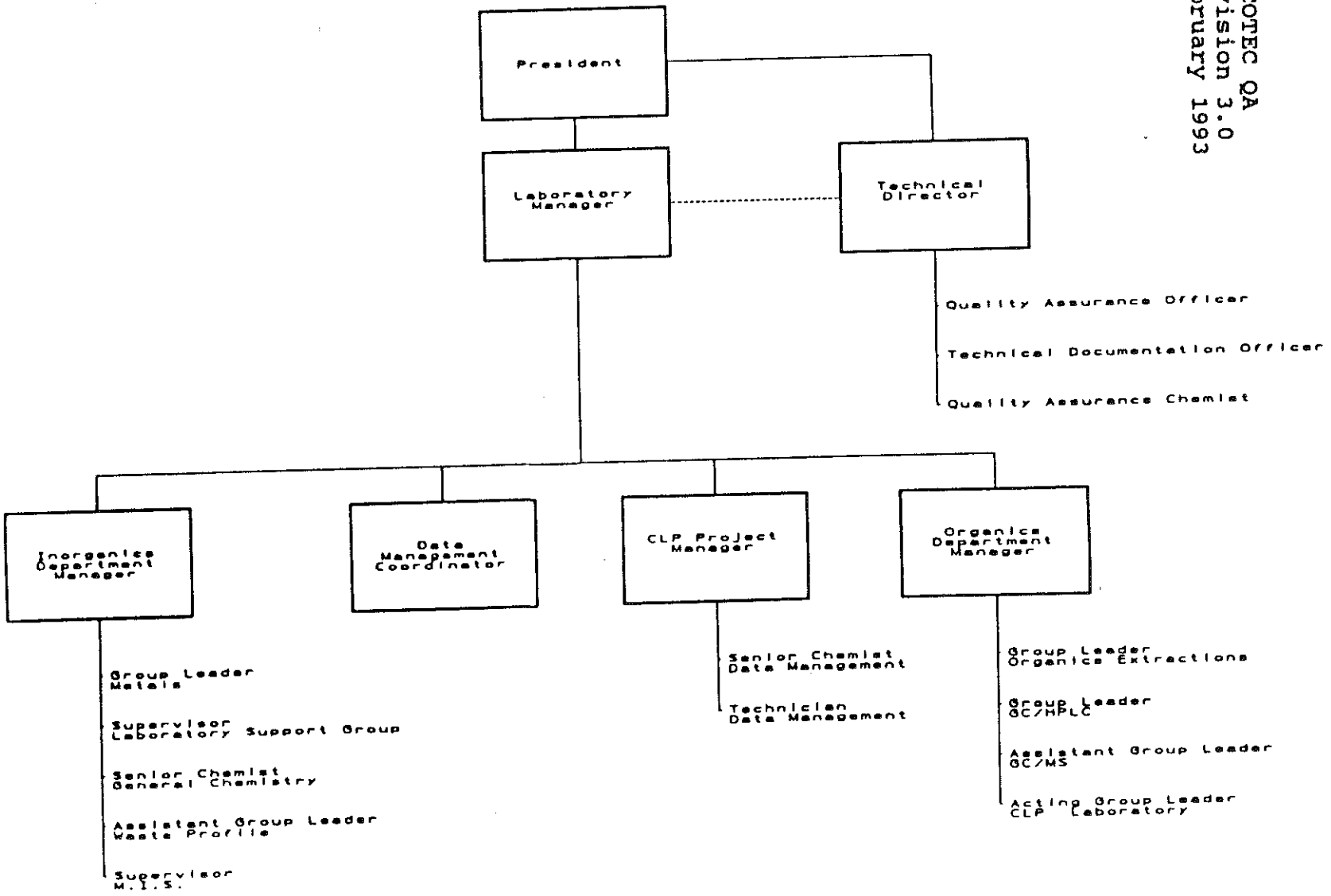


Fig. 4.2

Organics Department Organization

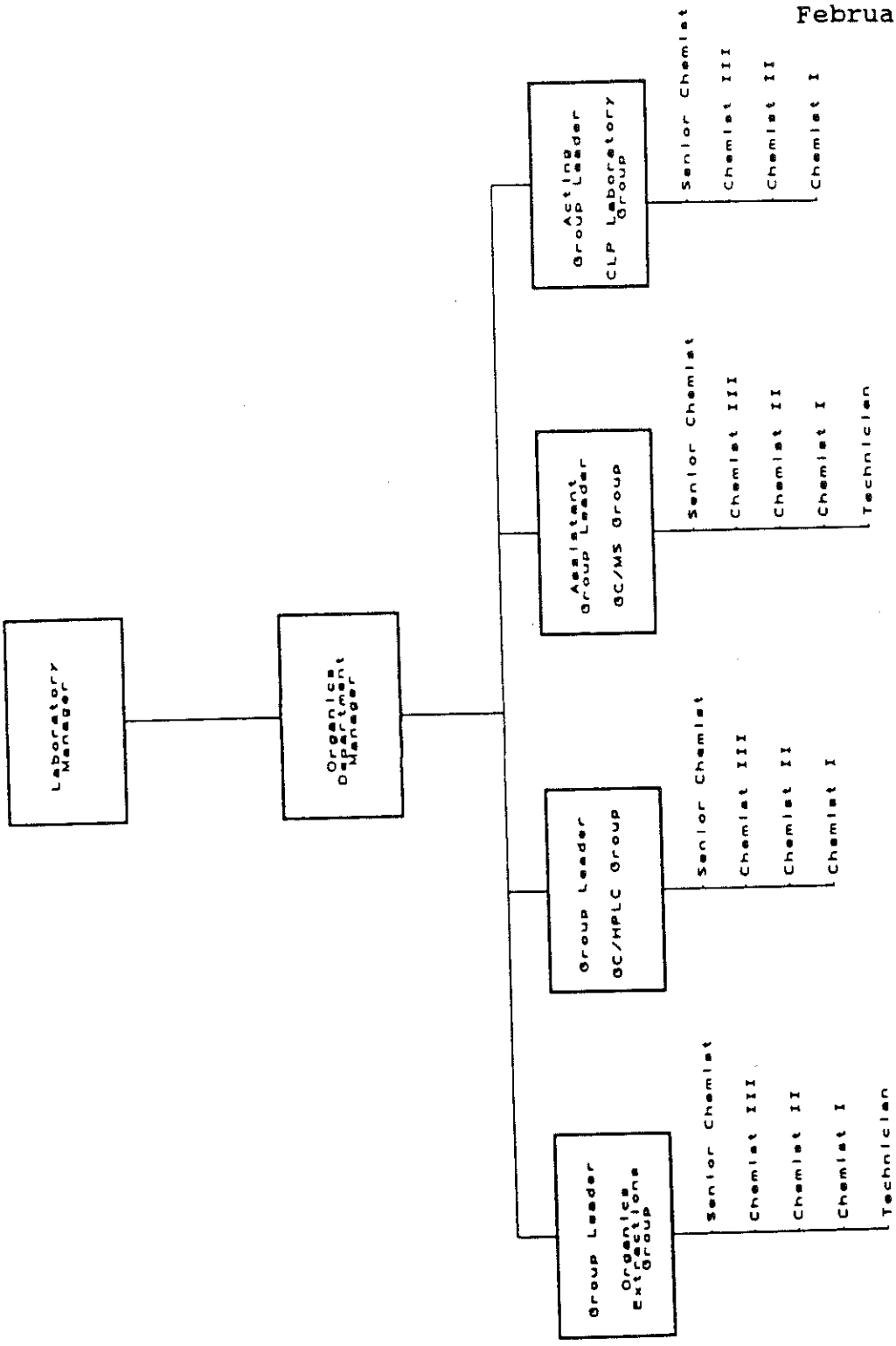
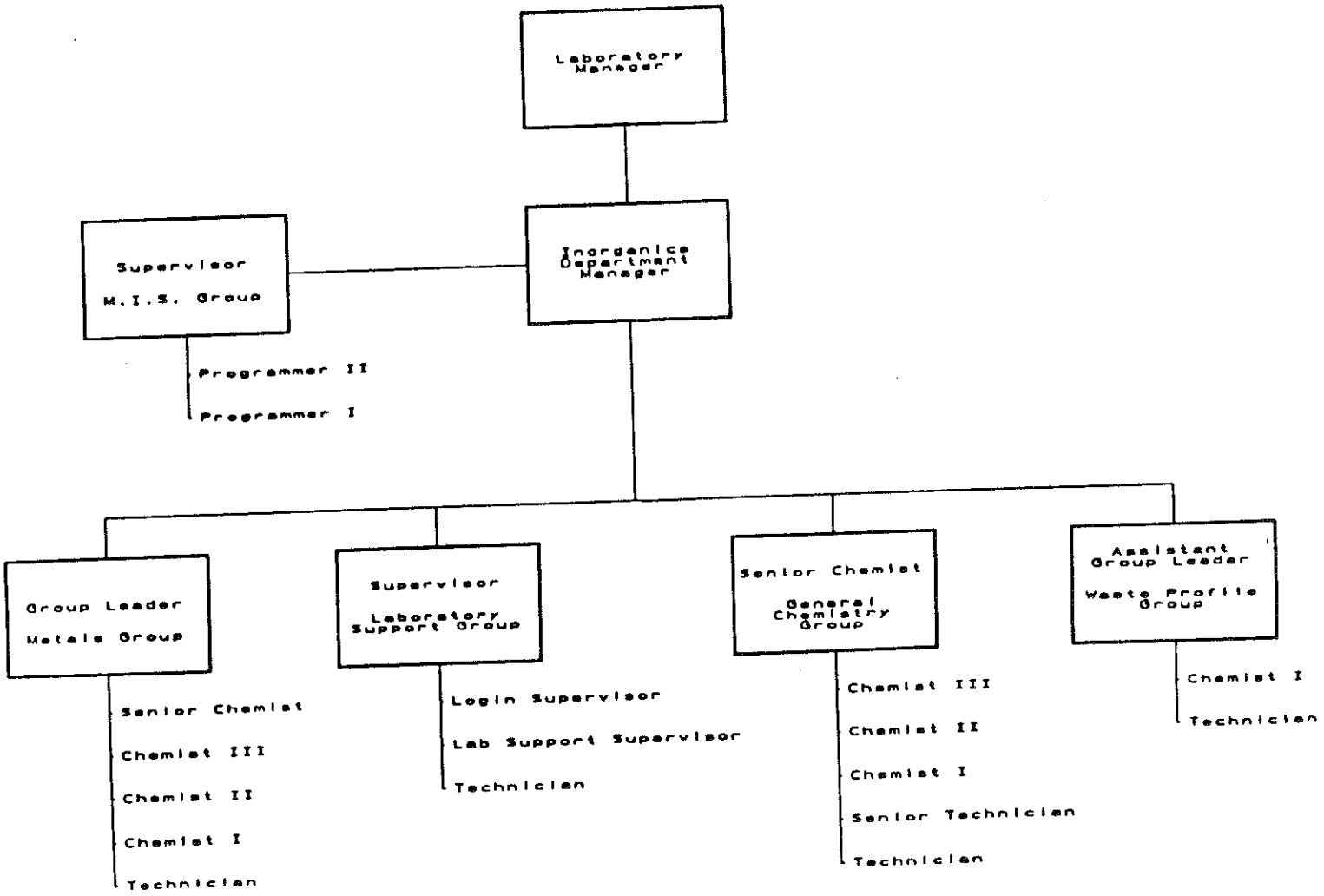


Fig. 4.3

Inorganics Department Organization



Quality Assurance Organization

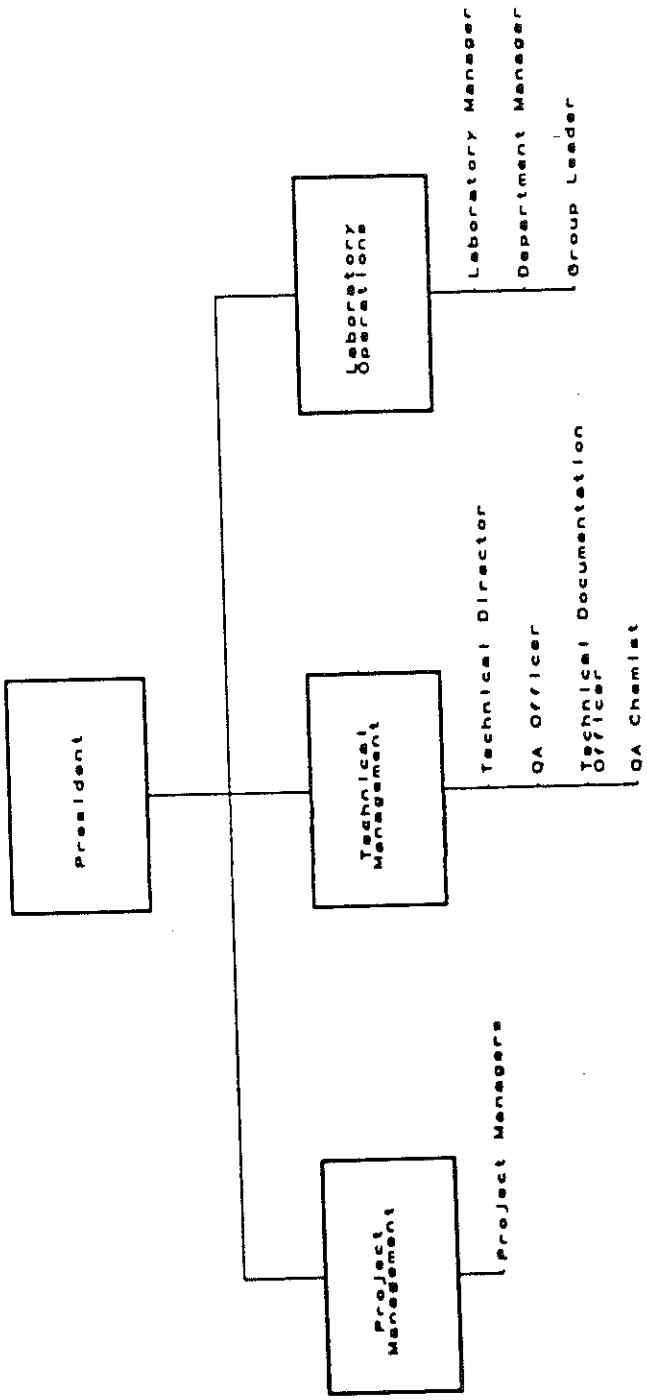


Fig. 4.5

The Technical Department is composed of the following individuals:

4.2.1 Technical Director

1) Provides overall technical direction to ENCOTEC's QA program, and laboratories.
2) Advises senior chemistry staff in the improvement of existing analytical methods, of agency approved changes in existing procedures, of proposed methodologies and in the resolution of difficult analytical problems.
3) Reviews and recommends changes in SOPs and company Quality Assurance Project Plans and coordinates new technical projects.

4.2.2 Quality Assurance Officer

1) Oversees current QC programs and further develops these programs, including QC data management.
2) Reviews and updates Quality Assurance Manual, develops Quality Assurance Project Plans (QAPP) and writes SOPs pertaining to quality control.
3) Conducts internal audits for the accurate assessment of laboratory and information producing practices and represents the company during external audits.
4) Works to expand certification for the laboratories under various state and federal programs and advises the company of state and federal agency quality assurance and quality control policy developments.

4.2.3 Technical Documentation Officer

1) Provides primary assistance in the writing and updating of technical documents and forms used primarily by the Chemistry Section, including review of Quality Assurance Project Plans.
2) Maintains document control and distribution of all SOPs and other administrative/procedural documents pertaining to quality control.
3) Transforms and formats operating procedures and other technical documents and/or forms provided by section groups into a format consistent with guidelines established by the Section.

4.2.4 Quality Assurance Chemist

1) Provides assistance in the implementation of current QC programs, including QC data management and review.
2) Conducts internal audits for the accurate assessment of laboratory and information producing practices.
3) Provides assistance in the writing and updating of technical documents and forms used primarily by the Chemistry Section, including SOPs.

4.3 Quality Control Management Structure

Implementation, on a routine daily basis, of quality control procedures within each department, occurs at the Group Leader through Senior Chemist level. Specific needs of each department

will dictate how the daily review of analytical data, including QC results, is managed. Laboratory group approval of data and QC information resides with the group leader of the respective laboratory. Review and assessment of quality control data resides with the Quality Assurance Officer. Senior chemists and staff chemists (I, II, and III) comprise the bulk of the analytical capability of the chemistry section. The responsibilities of these individuals, with respect to quality control, are to follow documented QC procedures and to recognize and diagnose, if possible, out-of-control analytical systems. Corrective action, if needed, is reported to the Quality Assurance Officer with implementation occurring at the supervisory level.

Figures 4.6 through 4.10 illustrate the organizational structure of each department within the chemistry section with respect to quality assurance practices.

Quality Control - Inorganics
Organization

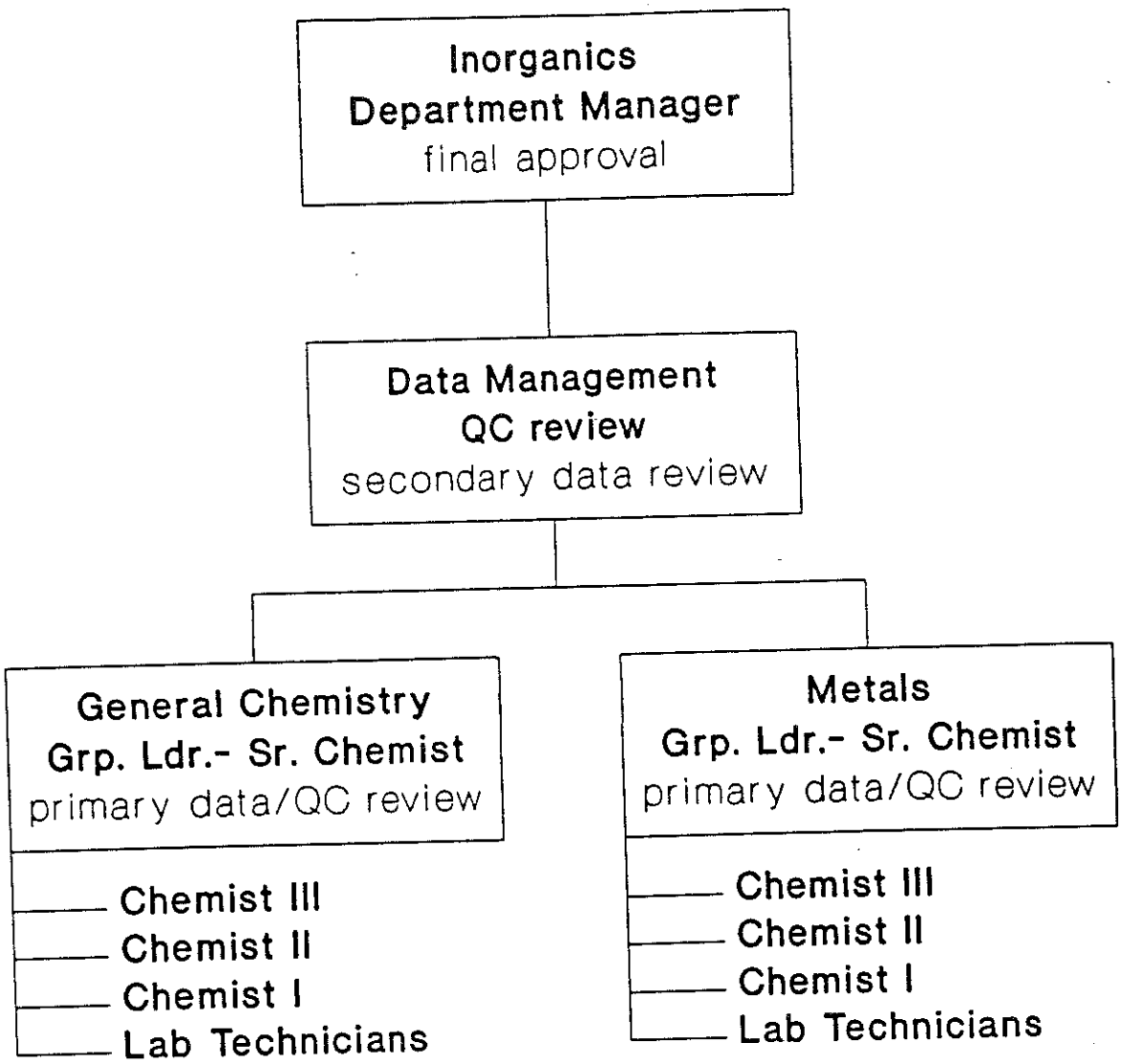


Fig. 4.6

Quality Control - GC/MS
Organization

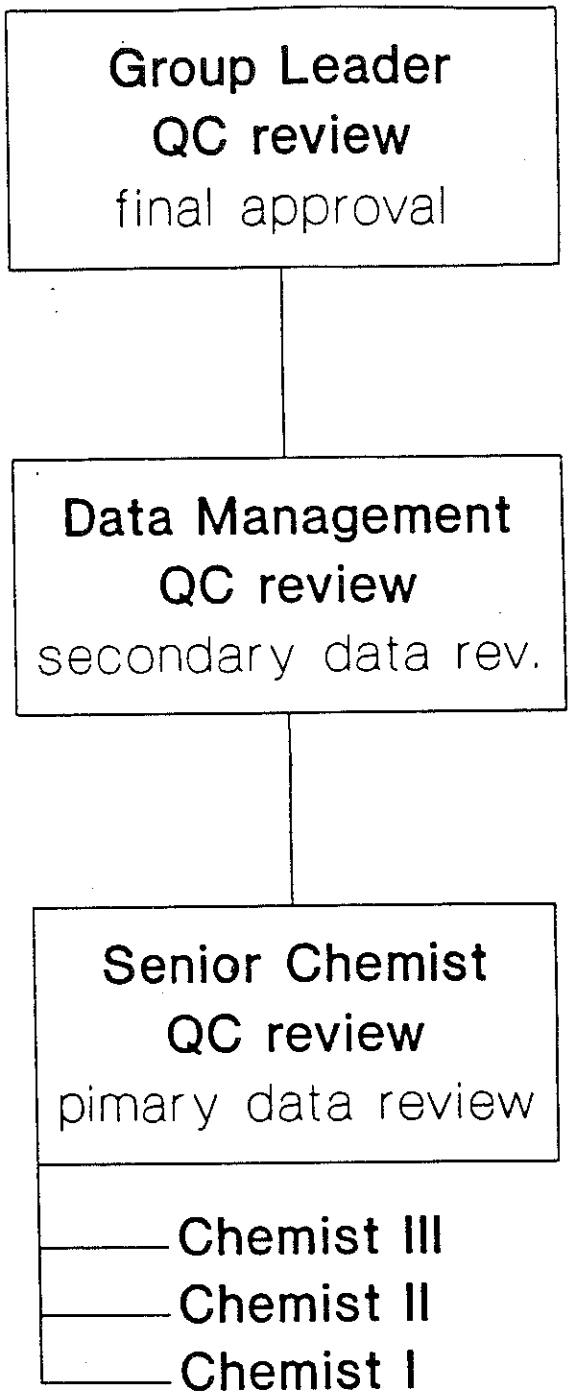


Fig. 4.7

Quality Control - GC/HPLC
Organization

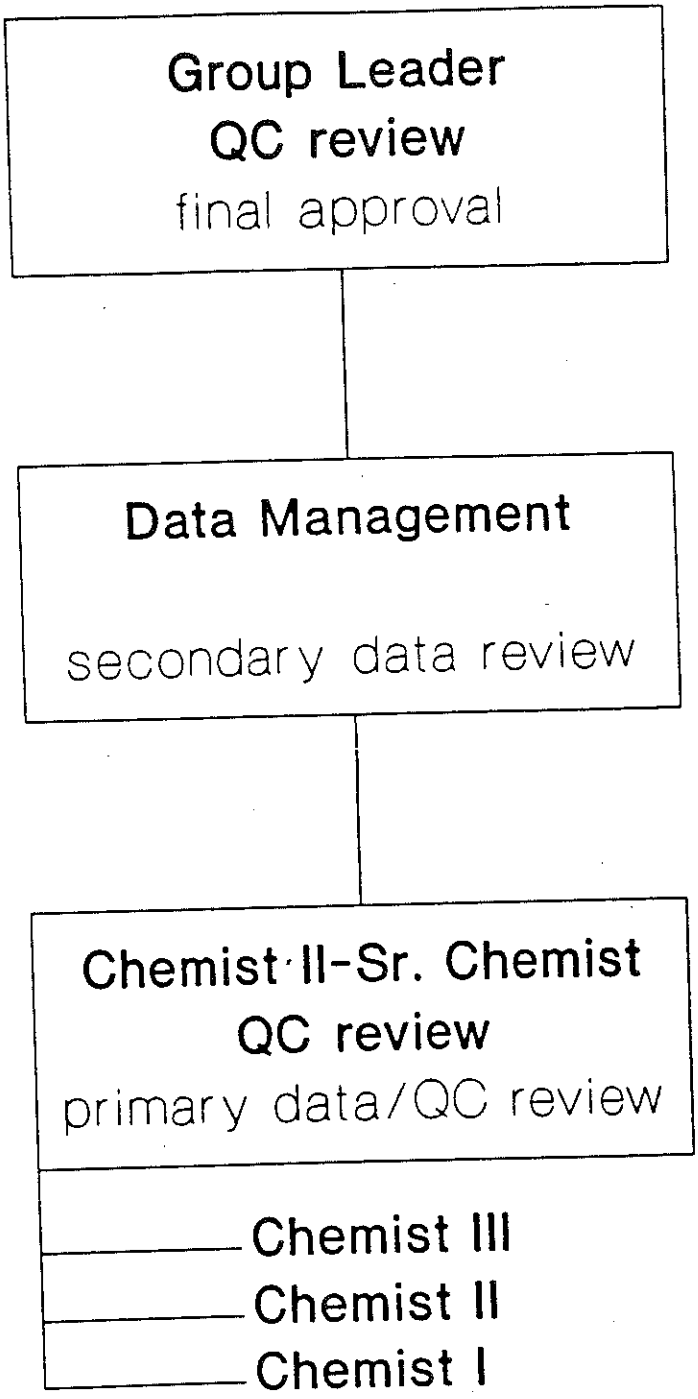


Fig. 4.8

Quality Control - Org. Extractions
Organization

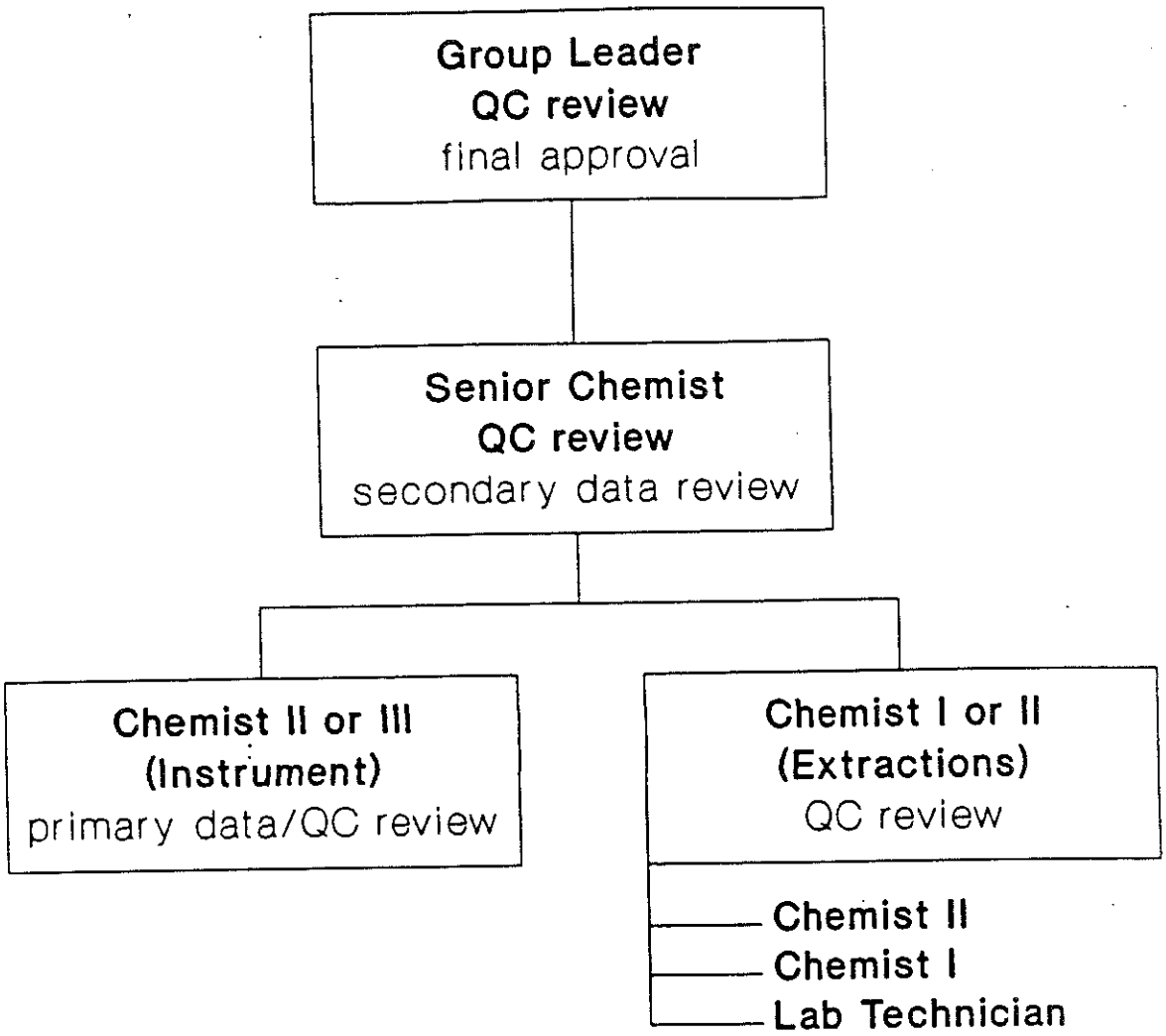


Fig. 4.9

Quality Control - Waste Profile
Organization

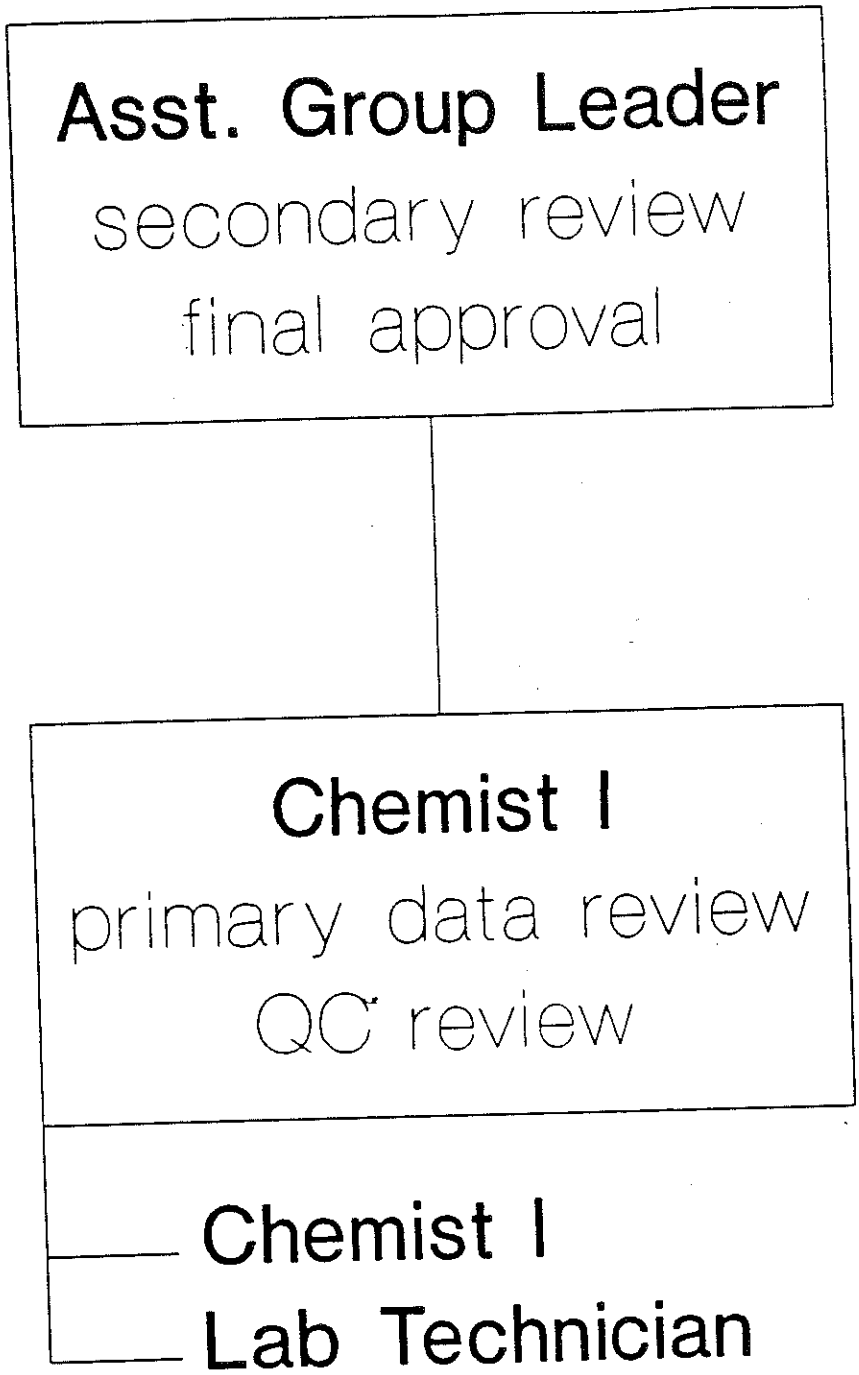


Fig. 4.10

5.0 ENCOTEC Employee Training

5.1 Introduction

Training is an integral aspect of a strong QA/QC program. It is imperative that an analyst be thoroughly trained on a given procedure before any analytical work is performed. Although entry level analysts are supervised and the raw analytical data is reviewed by experienced analysts, many potential problems can be avoided by proper training. Adequate training will help to ensure an appropriate introduction to the procedures employed for the particular analysis. It is important to correct poor laboratory techniques before negative impacts surface. At ENCOTEC, adequate training has proven to be the cornerstone to the success of the laboratory. Direct personal training by an experienced analyst provides the new analyst with the appropriate feedback needed to bring to light potential problem areas. This direct personal training also permits expedient corrective action. It is the goal of ENCOTEC to provide each analyst with a thorough introduction to the concepts and techniques involved in each analytical procedure.

5.2 ENCOTEC Minimum Requirements for Educational Training

Without the necessary educational experience, the most thorough training program will not ensure an adequate understanding of the concepts behind the analysis. To facilitate an effective training process, ENCOTEC maintains certain minimum criteria for the laboratory analyst addressing the basic academic background and experience required for employment. All ENCOTEC chemists possess a minimum of a bachelors degree in Chemistry (or related science),

and all technicians possess a minimum of two years of college experience with concentration in Chemistry (or related science).

5.3 Entry Level Analyst Training

All entry level analysts are exposed to a number of training programs. These initial training programs are designed to ensure the safety of the new analyst as well as the safety of those staff working side-by-side with the new analyst. The following are examples of these types of training programs:

a) OSHA Right-to-Know and Laboratory Safety Training

b) First Aid and CPR Training

c) ENCOTEC Procedures Training

5.4 Standard Operating Procedures

When an analyst is to be trained on a new method, the first step is to provide the analyst with a copy of the Standard Operating Procedure (SOP) and or the actual method reference. The SOP outlines the method step by step along with the QC requirements. Information is also provided regarding holding times, preservation requirements, and interferences.

5.5 Individualized Instruction

Individualized instruction is provided to the analyst-in-training by a group leader, senior chemist, or chemist III thoroughly knowledgeable in the procedure. The experienced chemist works with the novice outlining the chemistry on which the analysis is based, assists in the preparation and storage of standards, oversees the novice analyst's analysis of QC samples, and provides instruction in data reduction procedures and the calculation of QC results. This permits the analyst-in-training to perform the test with immediate feedback from the trainer. It also reinforces the novice chemist's understanding that there is an experienced individual in the laboratory group ready to assist.

5.6 Continuing Training

Continued training in any method with which an analyst has experience is as useful as the initial training the analyst receives, if not more so. As every analyst gains experience in an expanded number of methods, it is important to refine those skills associated with specific methods to ensure that results are accurately reported. When provided with additional education, supervision, and resources, analysts are able to expand both the breadth and depth of their experience to become training resources themselves.

5.7 Continuing Education

For a number of methods, particularly those which are instrument-intensive, ENCOTEC utilizes manufacturers' educational programs as a training resource. Analysts receive an education in instrument functions with an emphasis on maintenance, method development, and troubleshooting. This also presents a forum where the analyst can address the manufacturer directly. Posing and receiving answers to specific questions regarding the types of materials and the methods used while working with a given instrument helps to ensure that the equipment utilized in a given method performs as expected.

Many of these manufacturers also supply training videos that describe the instrument theory, function, and maintenance in sufficient detail that they can be used to train analysts.

Periodically, manufacturers provide technical seminars introducing advances or new instruments. Whenever possible, analysts are sent to gain a greater understanding of the instrumentation and to remain current on new developments.

5.8 Supervision

All analyses are performed under the supervision of a group leader, senior chemist, or chemist III within the laboratory. This provides an analyst with a ready resource for troubleshooting problems. The trainer provides guidance in scheduling samples for a given analysis and balancing priorities with sample holding time requirements. In addition, data sets are reviewed to ensure that errors are intercepted and all QA/QC requirements are met.

5.9 Resources

Copies of the SOPs for analyses performed by each laboratory group are available in the laboratory for reference by the analyst at any time. ENCOTEC purchases educational videos, when available, that introduce basic theory, practical applications, and history of environmental legislation, enhancing the analyst's understanding of tests and the purpose and use of the data. Videos are also generated in-house by ENCOTEC and its sister companies providing information pertaining directly to the company. A library of materials is also maintained covering environmental legislation, analytical chemistry methods, environmental engineering, previous projects, and health and safety information.

6.0 Facilities and Instrumentation

6.1 Introduction

Facilities and equipment are an often overlooked element of a well-functioning QA/QC program. In many ways a well-engineered and designed laboratory can be a very influential factor in producing data of high quality. ENCOTEC is well aware of the positive impact of its laboratory facilities and equipment on data quality and has always worked diligently to maintain the highest possible standards for instrumentation and for its laboratory facilities.

6.2 Space Allocation and Design Criteria

ENCOTEC has identified important criteria for the determination of space allocation and design of the laboratories. Thorough evaluation of each and every item has been made to ensure that the laboratory facilities are the finest possible, thus ensuring that when coupled with the other elements of this QA/QC manual, data of acceptable caliber can be produced. The following is a list of those criteria with a brief explanation of goals that have been achieved:

6.2.1 Adequate Floor Space

This criterion changes with sample loadings and the amount of physical space required to perform analyses properly. ENCOTEC provides approximately 12,000 square feet of floor space devoted to a variety of laboratory applications. It is very important to ensure that analysts work in an environment that is not over-crowded and that analysts have adequate space to perform their tasks.

6.2.2 Adequate Bench Space

This criterion can be evaluated with respect to the maximum number of analysts using any given laboratory and can be looked at in terms of a ratio. ENCOTEC guidelines are that every analyst on a given shift should have a minimum of 10 feet of linear bench space available to perform his/her work.

6.2.3 Adequate Hood Space

ENCOTEC has dedicated significant amounts of bench space to fume hoods for operations which require negative pressure (e.g. use of acids/solvents, preparing hazardous or potentially hazardous samples, or testing which evolves dangerous vapors/gases). ENCOTEC maintains over 160 linear feet of hood space, with over 49,000 cubic feet/minute of laboratory exhaust air. Most hoods are fitted with make-up air duct systems which supply fresh air from outside the building. Areas of high hood usage are under negative pressure.

6.2.4 Air Flow Balance

Laboratories which perform volatiles analysis should be under positive pressure to ensure that common laboratory solvents such as methylene chloride do not diffuse into those laboratories. All areas where solvent usage is high should be under negative pressure. ENCOTEC laboratories have been designed with this requirement in mind.

6.2.5 Adequate Power Requirements

A significant amount of time HAS BEEN DEVOTED TO determining and meeting electrical power requirements in the laboratories. The result is a system involving four major circuit types, supplying ample power to all laboratories. A 110V circuit for general usage, signified by white outlets, is available to all areas of the building. A separate set of "clean" 110V circuits, indicated by orange outlets, is dedicated for use by all computer-aided instruments/equipment. Single receptacle 110V circuits dedicated to analytical instrumentation are signified by brown outlets. Finally, all 220V single receptacle circuits dedicated to analytical instrumentation are signified by "twist lock" outlets.

6.2.6 High Purity Water

All laboratories are equipped with taps dispensing distilled and Super Quality water. These convenience taps ensure that all analysts have ready access to ample water of the purities specified for glassware washing and rinsing and for analytical use.

6.2.7 Clean Environment

ENCOTEC operates on a philosophy of "a clean laboratory will help to ensure quality". This cleanliness is important to reduce possibilities of reagent blank contamination. Dust removal is important to computer equipment. ENCOTEC has implemented a variety of management and design controls to ensure clean laboratories.

6.2.8 High Hazards Areas

ENCOTEC maintains areas for analysts to work with hazardous or potentially hazardous materials. Glove boxes and adequate hood space are integral to health threat minimization.

6.2.9 Information Access

All laboratories have ample access to the Laboratory Information Management Systems. A networked computer system is maintained for sample analysis scheduling. This information availability aids in producing compliant data within holding times. In addition, the system serves as an information collection device, storing testing results, raw chromatography data files and such interlaboratory information as sample pH and Percent Total Solids. The Laboratory Information Management System is maintained by the MIS staff and is archived regularly.

6.2.10 Data Review Areas

ENCOTEC has dedicated significant amounts of space to "quiet areas" for data review. These areas are equipped with computer stations for data handling. All applicable SOPs and reference materials are available.

The following blueprint facsimiles (Figures 6.1 and 6.2) are provided to detail the laboratory facility design and are referenced by Table 6.1 for information regarding area, linear bench space and linear hood space.

6.3 Instrumentation and Equipment

ENCOTEC maintains a significant array of state-of-the-art analytical instrumentation and equipment. While instrumentation and equipment lists do not ensure that quality services will be performed, an ample supply of these tools are needed to honor the copmany's commitments.

A variety of administrative controls is employed to determine when new instrumentation is necessary or to decide when to replace existing equipment. Logs of instrument usage, service requirements and corrective action are maintained and monitored to supply useful information to management. Five major guidelines pertain to all equipment and instrumentation:

1) All instrumentation/equipment should meet or exceed manufacturer's specifications.

2) All instrumentation/equipment should meet or exceed calibration criteria as required by the manufacturer.

3) All instrumentation/equipment is supplied with an operating environment consistant with manufacturer's specifications (i.e. humidity, temperature, air quality - including both chemical and physical needs, and proper water/electrical requirements).

4) All instrumentation/equipment is serviced on a routine basis, using "Preventative Maintenance" to ensure proper operation. This includes (where feasible) a system of formalized measurements of instrument performance, to serve as a warning of potential problems.

By implementing these guidelines ENCOTEC maintains the best possible instrumentation and equipment for the production of quality services. Appendix A, following, is a partial list of the equipment presently in use.

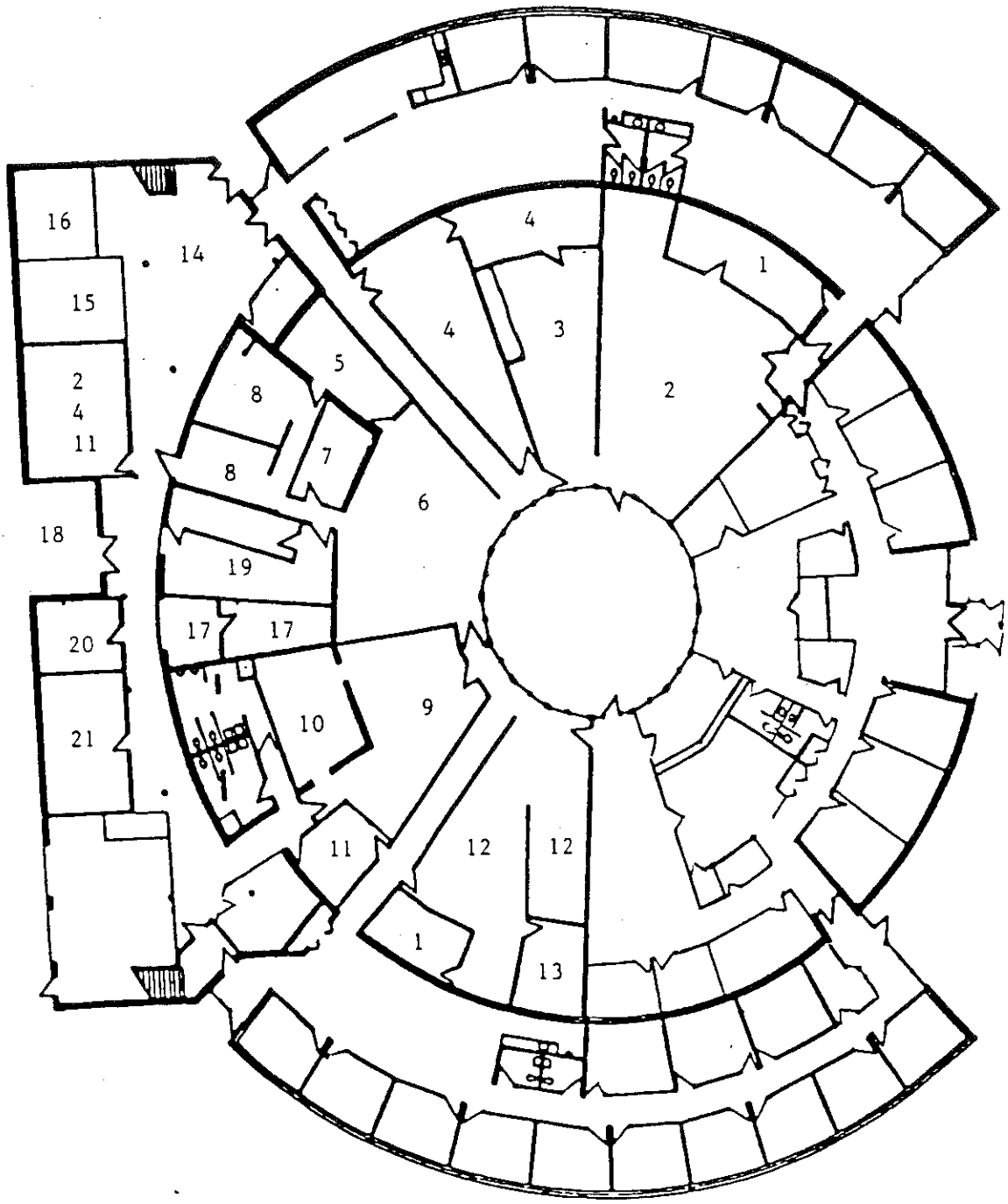


Figure 6.1: ENCOTEC I

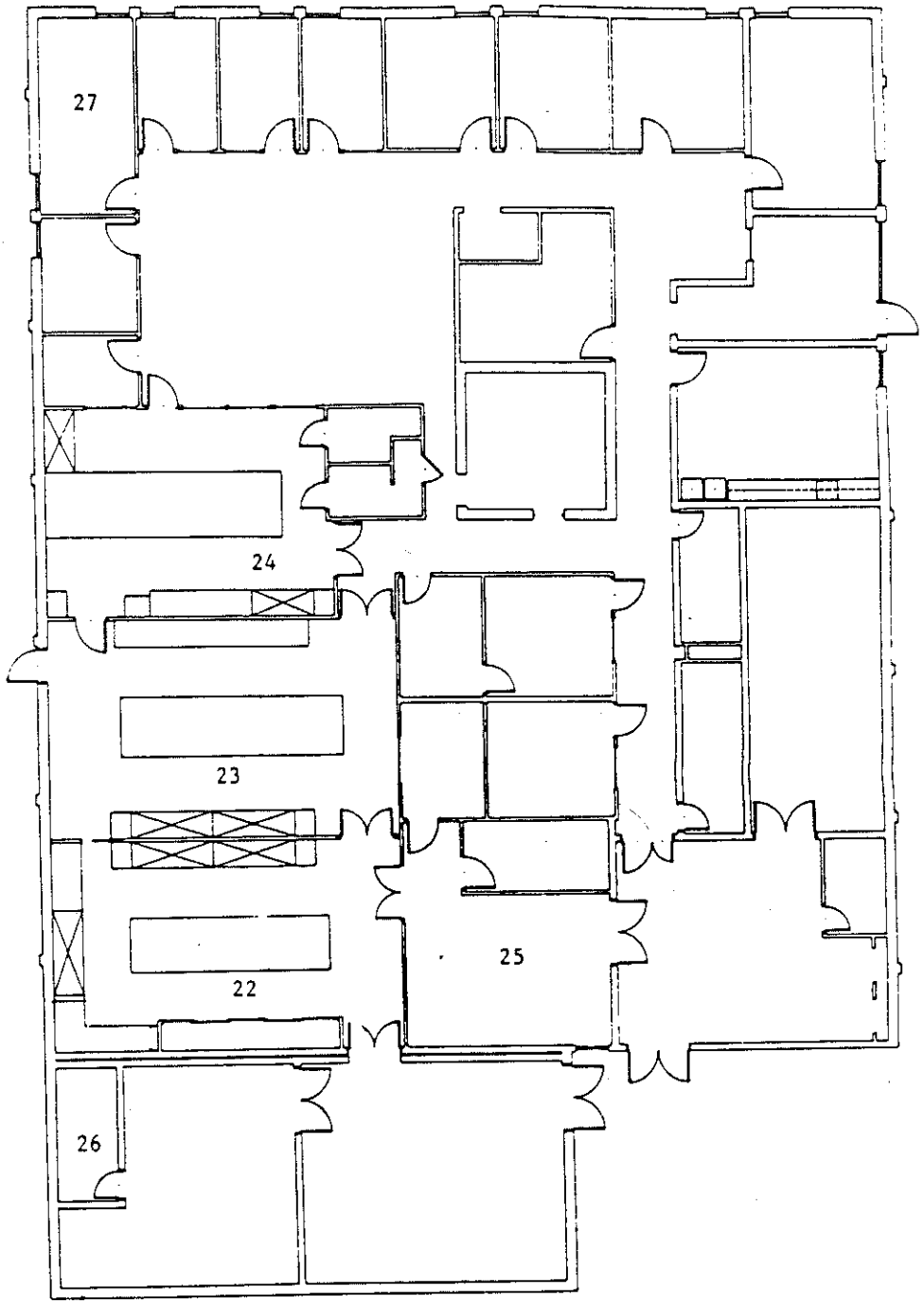


Figure 6.2: ENCOTEC II

TABLE 6.1
FACILITY SPACE ALLOCATION

Laboratory Number	Laboratory Description	Estimated Area (sq. ft.)	Bench Space (lin. ft.)	Hood Space (lin. ft.)
	ENCOTEC I, 3985 Research Park Drive			
1	Data Review Area	420	N/A	N/A
2	Inorganic Chemistry Laboratory	1035	130	42
3	Inorganic Extractions Laboratory	630	50	18
4	Metals Instruments Laboratory	910	53	0*
5	Standards Preparation Laboratory	250	21	6
6	GC/MS Volatiles Laboratory	770	59	0
7	Air Analysis Laboratory	160	14	N/A
8	GC/MS Semivolatiles Laboratory	620	31	0
9	Organic Extractions Main Laboratory	620	73	40
10	Organic Extractions Secondary Laboratory	320	45	27
11	Organic Extractions Instruments Laboratory	290	46	12
12	GC/HPLC Main Laboratory	960	88	12
13	GC/HPLC Volatiles Laboratory	190	24	0
14	Laboratory Support Group, Sample Receipt	400	40	4
15	Soil/Solid Sample Storage Walk-in Cooler	200	N/A	N/A
16	Water Sample Storage Walk-in Cooler	200	N/A	N/A
17	Sample Storage Area, Waste Samples	130	10	4
18	Gas Cylinder Storage Pad	270	N/A	N/A
19	Bottle Kit Preparation Laboratory	250	28	6
20	EE&A Treatability Laboratory	230	24	5
21	Bioassay Laboratory	445	38	0
	ENCOTEC II, 3965 Research Park Drive			
22	Waste Profile Laboratory	820	70	22
23	Organic Extractions CLP Laboratory	735	69	14
24	GC/GC/MS CLP Laboratory	520	58	9
25	Sample Receipt and Storage Area	300	N/A	N/A
26	Sample Storage Area, Waste Samples	62	N/A	N/A
27	Data Review Area	200	N/A	N/A
	Total Laboratory	11937	971	221

* Venting provided for instrument exhaust gases not included as available hoodspace.

7.0 SAFETY

Personnel and Laboratory Safety is a partnership between the employee and the company. ENCOTEC has promoted this partnership in many ways and has ensured its ongoing success through the ENCOTEC Safety Committee. Programs are designed with employee safety in mind and comply with all OSHA health standards outlined in 29 CFR Part 1910, Subpart 2 as it applies to the Laboratory Standard Application.

ENCOTEC promotes the continued development of five main tools to ensure employee safety:

7.1 Training and Educational Materials

All employees are required to attend training seminars taught by componay-provided specialists for:

1) First Aid/CPR
2) Laboratory Safety
3) Fire Extinguisher Use

In addition, a variety of supplemental training materials (books, manuals, videos) are available for review.

7.2 Right To Know Materials

All laboratory chemicals are itemized by location and current proper documentation is maintained and available.

7.3 Control Measures and Equipment

When it is necessary to work with dangerous or hazardous materials, health threat minimization is the goal. This goal is achieved through proper engineering and administrative measures. Implementation of procedure-specific safety procedures as well as the proper use of such devices as gloveboxes, hoods, respirators, face shields, lab coats, gloves and other safety devices ensure minimization of worker exposure.

7.4 Hazard Communication Program

As mentioned above, in certain instances or specific procedures, unusual safety precautions must be taken. These safety procedures are outlined in the Chemical Hygiene Plan.

7.5 Chemical Hygiene Plan

The Chemical Hygiene Plan is a written communication per 29 CFR 1910.1450, 7/92, of general standard operating procedures for safety within the laboratories. It addresses functional areas such as:

1) General Rules
2) Personal Hygiene
3) Food and Smoking
4) Protective Clothing and Equipment
5) Housekeeping
6) Spills and Accidents
7) Waste Chemicals
8) Storage Cabinets
9) Ventilation
10) Fire Guidelines
11) Medical Consultation
12) Documentation

The company is committed to providing a safe working environment for its employees. With the functional tools mentioned above, ENCOTEC can implement measures to ensure that this commitment is successful.

8.0 Preventative Maintenance

To assure the quality of the data produced, it is necessary to maintain both the instruments and the facilities that support the daily operations of the lab. During the normal operations of an analytical laboratory, some of this maintenance must be performed at regularly scheduled intervals even if the instrument is producing quality data. This is preventative maintenance. To accomplish this, each laboratory will incorporate a variety of methods depending upon the sophistication of the equipment or instrumentation used. Proper maintenance will improve the precision and accuracy of the analyses performed as well as increase laboratory production efficiency. Although the individual labs operate independently to achieve this goal, an overall company philosophy is embraced. The basic guidelines recognized by ENCOTEC for the maintenance of laboratory instrumentation are:

- Instruments must be cleaned and inspected on a regular basis, as well as after the analysis of particularly difficult samples. All activity is recorded in logs placed by the instrument or in the lab.

- Instrument performance is to be monitored, primarily, by an experienced analyst(s) responsible for the instrument. Group Leaders through Senior Chemists will monitor this activity to ensure that company standards are met and will assist analysts as needed.

- The analyst responsible for an instrument is also responsible for the instrument log.

- An adequate supply of consumable parts as well as additional hardware is available to ensure continued instrument operation.

- Control measures such as instrument performance checks are used to evaluate the need for maintenance.

Ongoing maintenance procedures are instituted in order to recognize a possible or probable instrument malfunction before it results in unsatisfactory performance. For those laboratories which require manufacturer assistance in instrument maintenance, service agreements which include regularly scheduled preventative maintenance are utilized.

9.0 Analytical Procedures

9.1 Methods

The analyses performed by ENCOTEC are based upon the requirements of a particular project. In general all ENCOTEC methods are based upon the following:

1. Test Methods for Evaluating Solid Waste, USEPA SW-846, Third Edition. Originating Federal program: Resource Conservation and Recovery Act. Air methods cited include: EPA "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air", 1986 and 1988 revisions.

2. USEPA Contract Laboratory Program (CLP) Organic and Inorganic Statements of Work (SOW), OLM01.0 through OLM01.8 (Organics) and ILM03.0 (Inorganics). Originating Federal programs: Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) and Superfund Amendments and Reauthorization Act (SARA).

3. USEPA Methods for Chemical Analysis of Water and Wastewater and Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, 3/83 and 10/84 Revisions, respectively. Originating Federal programs: Federal Water Pollution Control Act (FWPCA) and Safe Drinking Water Act (SDWA).

4. USEPA Methods for the Determination of Organic Compounds in Drinking Water, 12/88 Revision. Originating Federal program: Safe Drinking Water Act.

5. American Society for Testing and Materials (ASTM) - most current edition.

6. Standard Methods for the Examination of Water and Wastewater - published jointly by the American Public Health Association (APHA), American Water Works Associations (AWWA), and the Water Pollution Control Federation (WPCF) - most current edition.

7. For certain analyses, no equivalent EPA, Standard Methods, or ASTM procedure exists. These procedures are cited with an endnote number in the second column in the following tables.

Copies of methods and QC requirements are available in the laboratory and are also compiled in Quality Assurance Project Plans (QAPP) as required. The following Table 9.1 lists the various parameters and methods for most of the analyses performed at ENCOTEC. (Method reference numbers in these tables refer to the seven sources listed above.) Additional analyses and special detection limit requirements are addressed on a project-specific basis.

MAJOR ANALYTICAL METHODS

EPA approved methods, where possible, are used to generate analytical data for the following regulatory needs:

Compound List	Federal (State) Regulation
Priority Pollutant	NPDES
Hazardous Substance List	RCRA
Target Compound/Analyte List	Superfund
Appendix IX	RCRA
Toxicity Characteristic HSWA*	
Landban (1st, 2nd, and 3rd Thirds)	HSWA*
Michigan RCRA Metals	(Michigan DNR)
Act 64 and/or Act 307	(Michigan DNR)
TO-14 Compendium of Analyses	Clean Air Act

* Hazardous and Solid Waste Amendments to RCRA

TABLE 9.1
ANALYTICAL METHODS

Inorganic Parameters	Endnote	RCRA	CERCLA	NPDES	Standard Methods	Ambient Air
Acid scrub	1	NA	NA	NA	NA	NA
Acidity (pH)		NA	NA	305.1	NA	NA
Alkalinity (pH)		NA	NA	310.1	NA	NA
Ammonia nitrogen		NA	NA	350.1	NA	NA
Biochemical oxygen demand		NA	NA	405.1	NA	NA
Bromide (ISE)		NA	NA	NA	NA	NA
BTU	2	NA	NA	NA	NA	NA
Carbon (inorganic)		9060	NA	415.1	NA	NA
Carbon (total)		9060	NA	415.1	NA	NA
Chemical oxygen demand		NA	NA	410.4, 410.1	NA	NA
Chloride		9251, 9252	NA	325.2, 325.3	NA	NA
Chlorine (demand)		NA	NA	NA	4500-Cl B	NA
Chlorine (residual)		NA	NA	330.5	NA	NA
Chromium (hexavalent)		7196	NA	NA	3500-Cr D	NA
Conductivity		9050	NA	120.1	NA	NA
Corrosivity		1110	NA	NA	NA	NA
Cyanide (amenable)		9012	NA	335.1	NA	NA
Cyanide (reactive)		7.3.3.2	NA	NA	NA	NA
Cyanide (total)		9012	CLP	335.2	NA	NA
Flashpoint		1010	NA	NA	NA	NA
Fluoride (free)		NA	NA	340.2	NA	NA
Halogens (%)	3	NA	NA	NA	NA	NA
Hardness (Ca + Mg)		NA	NA	NA	2340 B	NA
Hardness (titrimetric)		NA	NA	130.2	NA	NA
Mercury		6010, 7470, 7471	CLP	245.1	NA	NA
Metals other than Mercury and Hexavalent Chromium		6010, 6020 7000 Series	CLP	200.7, Metals, 200 Series	NA	HVF (6010)
Nitrate nitrogen		NA	NA	353.2	NA	NA
Nitrate/Nitrite nitogen		NA	NA	353.2	NA	NA
Nitrite nitrogen		NA	NA	353.2	NA	NA
Nitrogen (total Kjeldahl)		NA	NA	351.2	NA	NA
Oil and grease		9070	NA	413.1	NA	NA
Paint filter test		9095	NA	NA	NA	NA
pH		9040, 9041, 9045	CLP	150.1	NA	NA
Phenols (4-AAP)		9065	NA	420.1	NA	NA
Phosphorus (ortho)		NA	NA	365.3	NA	NA
Phosphorus (total)		NA	NA	365.3	NA	NA
Potassium		6010	CLP	200.7	3500-K D	NA
Sodium		6010	CLP	200.7	3500-Na D	NA
Solids (total dissolved)		NA	NA	160.1	NA	NA
Solids (total suspended)		NA	NA	160.2	NA	NA
Solids (total volatile)		NA	NA	160.4	NA	NA
Solids (total %)		NA	CLP	160.3	NA	NA
Specific Gravity		NA	NA	NA	2710 E	NA
Sulfate		9038	NA	375.4	NA	NA
Sulfide (reactive)		7.3.4.2	NA	NA	NA	NA
Sulfide (total)		9030	NA	376.1	NA	NA
Sulfite		NA	NA	NA	4110 B	NA
Sulfur (%)	4	NA	NA	NA	NA	NA
Total Petroleum Hydrocarbons		NA	NA	418.1	NA	NA
Turbidity		NA	NA	180.1	NA	NA
Viscosity	5	NA	NA	NA	NA	NA

Abbreviation conventions:
CLP = Contract Laboratory Program Statement of Work.
HVF = High volume filter taken from the high volume air sampler.
NA = Not applicable.

See Endnotes for further details

TABLE 9.1 - CONTINUED
ANALYTICAL METHODS

Organic Parameter Groups	Endnote	RCRA	CERCLA	NPDES	Standard Methods	Ambient Air
Alcohols		8015	NA	NA	NA	NA
Total Petroleum Hydrocarbons	6	8015	NA	NA	NA	TO-13
Organic carbon (purgeable)	7	NA	NA	NA	NA	NA
Organic carbon (total)		9060	NA	415.1	NA	NA
Organic halogens (purgeable)	8	NA	NA	NA	NA	NA
Organic halogens (total)		9020, 9022	NA	NA	NA	NA
Semivolatile Organics						
Base/neutrals and acids		8270	CLP	625	NA	TO-13
Benzidines		8270	CLP	625	NA	TO-13
Chlorinated hydrocarbons		8270, 8120	CLP	625, 612	NA	TO-13
Dioxins and dibenzofurans		8270	NA	NA	NA	NA
Haloethers		8270	CLP	625	NA	TO-13
Nitroaromatics and isophorone		8270	CLP	625	NA	TO-13
Nitrosoamines		8270	CLP	625	NA	TO-13
OCL herbicides		8150	NA	NA	6640 B	TO-4
OCL pesticides		8080	CLP	608	NA	TO-4
OP pesticides		8140	NA	NA	NA	TO-4
PAH's		8270, 8310, 8100	CLP	625, 610	NA	TO-13
PCB's		8080	CLP	608	NA	TO-4
Phenols		8270, 8040	CLP	625, 604	NA	TO-13
Phthalate esters		8270, 8060	CLP	625, 606	NA	TO-13
Volatile Organics						
Acrolein	9	8240, 8260, 8030	CLP	624, 603	NA	TO-1, TO-2, TO-14
Acrylonitrile	9	8240, 8260, 8030	CLP	624, 603	NA	TO-1, TO-2, TO-14
Haloethers	9	8240, 8260, 8010	CLP	624, 601	NA	TO-1, TO-2, TO-14
Ketones	9	8240, 8260, 8015	CLP	624	NA	TO-1, TO-2, TO-14
Nitriles		8240, 8260, 8030	CLP	624, 603	NA	TO-1, TO-2, TO-14
Purgeable aromatics	9	8240, 8260, 8020	CLP	624, 602	NA	TO-1, TO-2, TO-14
Purgeable chlorinated	9	8240, 8260, 8010	CLP	624, 601	NA	TO-1, TO-2, TO-14
THM's	9, 10	8240, 8260, 8010	NA	624, 601	NA	NA

Abbreviation conventions:
CLP = Contract Laboratory Program Statement of Work.
HVF = High volume filter taken from the high volume air sampler.
NA = Not applicable.

See Endnotes for further details

TABLE 9.1 - CONTINUED
ANALYTICAL METHODS

Sample Preparation, Extraction and Cleanup	Endnote	RCRA	CERCLA	NPDES	Standard Methods	Ambient Air
Cleanup (alumina column)		3610	NA	NA	NA	NA
Cleanup (florisil column)		3620	CLP	NA	NA	HVF (3620)
Cleanup (gel permeation chromatography-GPC)		3640	CLP	NA	NA	NA
Cleanup (silica gel column)		3630	NA	NA	NA	NA
Cleanup (sulfur)		3660	CLP	NA	NA	HVF (3660)
Digestion (flame AA or ICP AES)		3010, 3050	CLP	200.7	NA	HVF (3050)
Digestion (furnace AA)		3020, 3050	CLP	200.7	NA	NA
Extraction (semivolatiles)		3510, 3520, 3540, 3550	CLP	NA	NA	HVF (3540)
Extraction (purge and trap)		5030	CLP	NA	NA	NA
Leaching procedure (EPTOX)		1310	NA	NA	NA	NA
Leaching procedure (TCLP)		1311	NA	NA	NA	NA
Leaching procedure (soil leachate)	11	SM	NA	NA	NA	NA
Waste dilution (high level semi-volatile organics in any matrix)		3580	NA	NA	NA	NA
Waste dilution (high level volatile organics in any matrix)		3580	NA	NA	NA	NA

Endnotes:

1) Acid scrub performed on the caustic scrub water created in the BTU procedure.
2) Bomb calorimeter methods per manufacturer's recommendations. No equilvalent EPA, SM or ASTM procedure published.
3) % Total Halogen is performed on the caustic scrub water created in the BTU procedure.
4) % Total Sulfur is performed on the caustic scrub water created in the BTU procedure.
5) Viscosity methods per manufacturers' recommendations. No equivalent EPA, SM, or ASTM procedure published.
6) Reference method is ASTM D3328.
7) Modification of Total Organic Carbon analysis. Detection by IR. No equivalent EPA, SM or ASTM procedure.
8) Modification of the Total Organic Carbon analysis. Detection by ELCD (electrolytic conductivity detector). No equivalent EPA, SM, or ASTM procedure.
9) Reference method for drinking water is EPA 524.2.
10) Reference method for drinking water is EPA 501.1.
11) This procedure is a soil-to-distilled water leach of the constituents of concern. The results of analysis are expressed as "water-leachable" constituents.

Abbreviation conventions:
CLP = Contract Laboratory Program Statement of Work.
HVF = High volume filter taken from the high volume air sampler.
NA = Not applicable.

9.2 Reagents and Traceability

The purity of all chemicals and reagents meets the following minimum standards:

All chemicals used in the laboratories are ACS reagent grade quality or better. Solvents are distilled in glass, reagent grade and suitable for all trace organic and pesticide residue analysis.

Gases used in the laboratory vary in purity according to use requirements. Zero-grade compressed air, prepurified acetylene, and prepurified argon are used for atomic absorption/emission spectrophotometry. The carrier gas for all gas chromatographs is ultra-high purity nitrogen, helium, or hydrogen. Prepurified nitrogen is used for extract concentration.

All chemicals are dated upon receipt and are used on a first in - first out basis. Analysts monitor chemical "age" to ensure only acceptable materials are used.

Certain reagents and all analytical standards require validation checks. Standards are proven traceable to NIST or EPA reference materials. The analytical procedures for standards validation are presented in the Standards Preparation SOPs.

Solvent, gas, water, reagent, etc. purity is monitored by checking results of analysis of method blanks carried through all preparation and analysis procedures with each batch of samples. Criteria defining method blank contamination are established in each method.

All critical volume determinations employ class "A" volumetric glassware, including pipets and volumetric flasks, etc. Samples and reagents are stored in clearly labeled and dated polyethylene or glass bottles.

Laboratory grade ASTM Type II water is produced by a deionization process, followed by filtration through specific-sized screens. Using activated carbon, cation/anion and mixed bed resin tanks, impurities and particulate matter are removed from the water. A Millipore Super Quality system is used to further clean and polish the water so that a resistivity of at least 10 megohms is reached. A SQ/DI water quality log book is maintained and samples taken at random from the different locations in the building are tested monthly for chlorine and chloride, bi-weekly for volatiles, and weekly for conductance. Preventative maintenance includes replacing the tanks and filters at predetermined intervals and disinfecting the system twice a year.

9.3 Calibration Procedures

Calibration procedures are defined in each method used in the laboratory. These procedures meet or exceed the requirements of the specific EPA methods being utilized. Daily calibration includes initial and continuing calibration verification, standards reference checks using an independent standard, and blank analyses. Specific levels for five-way calibration and initial and continuing calibration verification, as well as other information, are described in the individual methods and Standard Operating Procedures.

10.0 Sample Handling

10.1 Sample Receipt

When samples are delivered to the environmental laboratory, they are recorded in a Master Logbook and given a sequential laboratory number. The company name, facility and date received are recorded in the logbook. A laboratory report form/receipt is recorded in the logbook. A laboratory report form/receipt document is then filled out for each set of samples, recording the lab number, client name, facility, sample identification field data, date received and analysis requested. All paperwork which arrived at the laboratory with the samples , such as purchase orders, chain-of-custody documents, notes from the clients, etc. are attached to the report form/receipt document. Any discrepencies are resolved via communication with the client before analytical work begins.

10.2 Chain of Custody

To assure custody of samples during transport and shipping, each sample or set of samples, is recorded on a numbered chain-of-custody record. The sampling locations as well as the date and time of collection and analysis parameters requested are recorded. To complete the chain of possession, the collector will sign and date the form as will all those involved in the chain of possession. Samples are received at the laboratory by the Laboratory Support Group staff. Integrity of samples is verified and the chain-of-custody record signed and dated. The original custody form is then filed in the project file.

The samples received at the laboratory will be handled according to the standard operating procedure for samples. Sample tracking and record keeping are described in the SOP.

Sample preservation entails refrigeration at 4°C after preservation, where applicable. All samples except those designated for metals and/or waste profile analysis are refrigerated upon receipt. Volatiles are stored in a separate refrigerator. Extracts are stored in an extract refrigerator at 4°C until analyzed. All refrigerator temperatures are checked daily. The attached tables shows the current preservation and holding time requirements for various analyses.

10.3 Sample Preservation and Holding Times

- See Table 10.1 which follows.

TABLE 10.1
HOLDTIME, SAMPLE VOLUME AND PRESERVATION REQUIREMENTS

Method Description	Reference Method(s)	Holding Time	Minimum Sample Required Soil / Water	Preservation Requirements Chemical	Physical
Acidity	305.1	14 Days	100 mL	None required	4 degrees C
Alkalinity	310.1	14 Days	100 mL	None required	4 degrees C
Biochemical Oxygen Demand	405.1	48 Hours	1 Liter	None required	4 degrees C
Bromide	MR	28 Days	100 mL	None required	4 degrees C
Carbon, Total Inorganic and/or Organic	9060 415.1	28 Days	10 g / 25 mL	pH < 2, H2SO4 or HCl	
Chemical Oxygen Demand	410.4, 410.1	28 Days	50 mL	pH < 2, w/H2SO4	4 degrees C
Chloride	9251, 9252 325.2, 325.3	28 Days	50 mL	None required	None required
Chlorine (Total residual)	330.5	Analyze immediately	200 mL	None required	None required
Corrosivity toward Steel	1110	None specified	500 mL	None required	Glass vessel
Cyanide, Total and/or Amenable	9012 335.2	14 Days	10 g / 500 mL	pH > 12, w/NaOH Add 0.6 g Ascorbic Acid if Cl2 present	None required
Flashpoint, closed-cup	1010	None specified	200 g	None required	None required
Fluoride	340.2	28 Days	300 mL	None required	Plastic vessel
Hardness	130.2, 2340 B	6 Months	500 mL	pH < 2 w/HNO3	4 degrees C
Hexavalent Chromium	7196, 3500-Cr D	24 Hours	10 g / 100 mL	None required	4 degrees C
Mercury, Aqueous Matrices	7470, 245.1	28 Days	10 g / 200 mL	pH < 2, w/HNO3	None required
Mercury, Soil/Solid Matrices	7471	28 Days	10 g / 200 mL	pH < 2, w/HNO3	None required
Metals Analysis, other than Mercury and Hexavalent Chromium	6010, 6020, 7000 series Metals, 200.7, 200 series	6 months	10 g / 200 mL	pH < 2, w/ HNO3	None required
Nitrogen as Ammonia	350.1	28 Days	500 mL	pH < 2, w/H2SO4	4 degrees C
Nitrogen, Total Kjeldahl	351.2	28 Days	500 mL	pH < 2, w/H2SO4	4 degrees C
Nitrogen as Nitrate or Nitrite	353.2	48 Hours	100 mL	None required	4 degrees C
Oil and Grease	9070, 413.1	28 Days	10 g / 1 Liter	pH < 2, w/H2SO4	Glass vessel, 4 deg. C
Paint Filter Test	9095	None specified	200 g	None required	None required
pH (Electrometric or paper)	9040, 9041, 9045, 150.1	Analyze immediately	100 g / 25 mL	None required	None required
Phenolics (4-AAP)	9065, 420.1	28 Days	500 mL	pH < 2, w/H2SO4	Glass vessel, 4 deg. C
Phosphorus as Orthophosphate	365.3	48 Hours	50 mL	pH < 2, w/H2SO4	4 degrees C
Phosphorus, total	365.3	28 Days	50 mL	pH < 2, w/H2SO4	4 degrees C
Reactive Cyanide	7.3.3.2	14 Days	200 g	None required	Glass vessel
Reactive Sulfide	7.3.4.2	14 Days	200 g	None required	Glass vessel
Residue, Filterable	160.1	7 Days	100 mL	None required	4 degrees C
Residue, Non-filterable	160.2	7 Days	100 mL	None required	4 degrees C
Residue, Settleable	160.5	48 Hours	1 Liter	None required	4 degrees C
Residue, Total	160.3	7 Days	100 mL	None required	4 degrees C
Residue, Volatile	160.4	7 Days	100 mL	None required	4 degrees C
Specific Conductance	9050, 120.1	28 days	100 g / 100 mL	None required	4 degrees C
Sulfate	9038, 375.4	28 Days	50 mL	None required	4 degrees C
Sulfides	9030, 376.1	7 Days	100 g / 500 mL	Add Zinc Acetate pH > 9, w/NaOH	None required
Total Petroleum Hydrocarbons	418.1	None specified	200 g / 2 X 1 Liter		4 degrees C
Turbidity	180.1	48 Hours	100 mL	None required	4 degrees C

TABLE 10.1 - CONTINUED
HOLDTIME, SAMPLE VOLUME AND PRESERVATION REQUIREMENTS

Method Description	Reference Method(s)	Holding Time Soils / Waters	Minimum Sample Required Soils / Waters	Preservation Requirements Chemical	Physical
Halogenated Volatile Organics - GC	8010 601	14 Days	15 g / 2 X 40 mL	4 drops HCl (Waters only)	Glass vessel, 4 deg. C no headspace
Nonhalogenated Volatile Organics - GC	8015	14 Days	15 g / 2 X 40 mL	4 drops HCl (Waters only)	Glass vessel, 4 deg. C no headspace
Aromatic Volatile Organics - GC	8020, 602	14 Days	15 g / 2 X 40 mL	4 drops HCl (Waters only)	Glass vessel, 4 deg. C no headspace
Acrolein, Acrylonitrile Acetonitrile - GC	8030, 603	14 Days	15 g / 2 X 40 mL	4 drops HCl (Waters only)	Glass vessel, 4 deg. C no headspace
Volatiles - GC/MS	8240, 8260 624, 524.2	14 Days	15 g / 2 X 40 mL	4 drops HCl (Waters only)	Glass vessel, 4 deg. C no headspace
Total Organic Halides	9020 9022	7 Days	250 mL	< pH 2, w/H2SO4	Amber glass vessel, 4 deg. C, no headspace
Phenols	8040, 604	14 / 7 Days (extraction) 40 Days (analysis)	30 g / 2 X 1 Liter	None required	Amber glass vessel 4 degrees C
Phthalate Esters	8060, 606	14 / 7 Days (extraction) 40 Days (analysis)	30 g / 2 X 1 Liter	None required	Amber glass vessel 4 degrees C
Chlorinated Pesticides/PCB's	8080, 608	14 / 7 Days (extraction) 40 Days (analysis)	30 g / 2 X 1 Liter	None required	Amber glass vessel 4 degrees C
Polynuclear Aromatic Hydrocarbons	8100, 610	14 / 7 Days (extraction) 40 Days (analysis)	30 g / 2 X 1 Liter	None required	Amber glass vessel 4 degrees C
Chlorinated Hydrocarbons	8120, 612	14 / 7 Days (extraction) 40 Days (analysis)	30 g / 2 X 1 Liter	None required	Amber glass vessel 4 degrees C
Organophosphorus Pesticides	8140	14 / 7 Days (extraction) 40 Days (analysis)	30 g / 2 X 1 Liter	None required	Amber glass vessel 4 degrees C
Chlorinated Herbicides	8150, 6640 B	14 / 7 Days (extraction) 40 Days (analysis)	30 g / 2 X 1 Liter	None required	Amber glass vessel 4 degrees C
Semivolatiles - GC/MS	8270, 625	14 / 7 Days (extraction) 40 Days (analysis)	30 g / 2 X 1 Liter	None required	Amber glass vessel 4 degrees C
EPTOX extraction	1310	None specified	100 g	None required	Glass vessel
TCLP extraction	1311	14 to 180 days to extraction dep. on analysis	100 g	None required	Glass vessel
Volatiles on Carbon Sorbent	TO-1	14 Days	6000 cu. m	None required	4 degrees C
Volatiles on Tennax Sorbent	TO-2	14 Days	6000 cu. m	None required	4 degrees C
Pesticides/PCB's on Hi-Vol PUF/Filters, by GC	TO-4	14 / 7 Days (extraction) 40 Days (analysis)	300 cu. m	None required	4 degrees C
Semivolatiles on Hi-Vol PUF/Filters, by GC/MS	TO-13	14 / 7 Days (extraction) 40 Days (analysis)	300 cu. m	None required	4 degrees C
Volatiles in Summa Canister	TO-14	14 Days	6000 cu. cm	None required	None required
Metals/Particulates on Hi-Vol Filters	NA	28 Days to 6 Months	2000 cu. cm	None required	None required

11.0 Document and Forms Management

11.1 Introduction

Laboratory document and forms management procedures are in place to provide objective evidence of the work performed. The procedures outline specific details of the requirements and responsibilities for the generation, validation, distribution, retention, maintenance, safekeeping, and disposition of all documents and forms which can be associated with any sample received for analysis. The procedures help to ensure that ENCOTEC's records are legible, identifiable, retrievable and maintained in accordance with ENCOTEC management good laboratory practices guidelines.

11.2 Document Control

A system of documentation exists in order to provide a written record of all aspects of sample handling, analysis, computations, standard operating procedures, etc. This system includes the preparation of SOPs for the various aspects of the project including detailed descriptions of the various forms required to meet method requirements and the daily procedures to be followed.

ENCOTEC's staff includes a Technical Documentation Officer. Research and consultion with Senior Chemists (within the area of expertise) results in the formulation of a draft procedure. This precursor to a Standard Operating Procedure is widely distributed to the appropriate technical, laboratory management, QA/QC and laboratory personnel, who review the draft and submit comments. Once comments are received, included or rejected, and the

appropriate approval signatures are obtained, the document considered final. The Technical Department maintains both computer files of the SOPs for future revisions and the original signed copy of the Standard Operating Procedure. Standard Operating Procedures are distributed according to the ENCOTEC Document Control SOP. Logs are maintained for all released "controlled" copies of SOPs. If revisions to SOPs are made, the procedures described in the ENCOTEC Standard Operating Procedure for the Control of Method Manuals and Standard Operating Procedures are followed for all copies of the "controlled" SOPs in current use. It is an ENCOTEC requirement that all SOPs in use be "controlled" copies produced by technical management personnel.

The procedures employed for the formulation and promulgation of many Admininistrative Policies closely follow those described for SOPs.

The procedures employed for the formulation of Safety Policies also closely follow the those employed for formulation of SOPs. One minor exception is that the Chemical Hygiene Officer is the document control coordinator.

12.0 Data Reduction, Validation, Review and Reporting

12.1 Data Reduction and Assembly

In general, data reduction and assembly mechanisms vary with each analytical laboratory due to the differences in analytical instrumentation and methods used to generate analytical data. However, all analytical laboratories share certain similarities allowing specified minimum requirements for data reduction and assembly to be met. These requirements are discussed below.

12.1.1 - The analyst performing the test records all instrument conditions, calibration data, sample results, and quality control sample results on a laboratory bench sheet or computer log file and checks to ensure that all method and quality control criteria have been met. This retained information must be sufficient so that, should it be necessary, the analysis may be repeated or the analytical chain of events reconstructed.

12.1.2 - The analyst organizes the data package in a pre-defined format (specific to each laboratory) and then signs and dates the package before submission to data review personnel. A data organization sheet is used as an aid in compiling the data package elements as an easily reviewed document and in requiring the analyst to submit a standardized and complete data package. If the data package is non-compliant for any reason, the analyst records the reason(s) on laboratory specific forms. The data organization sheet becomes the data package cover page and is maintained as a formal and permanent document in the data file.

12.1.3 - A data management staffperson, most frequently a Chemist II, Chemist III, or Senior Chemist, provides primary review of the analytical data to ensure that all data package elements are present. The reviewer evaluates quality control data against ENCOTEC and reference method QC limits. He/she performs spot checks on analytical data, proofreads, and may transcribe and summarize the data. Any discrepancies are noted and recorded. If minor flaws exist, the reviewer will discuss and pursue resolution of the problem with the analyst. If major data flaws exist, discussion with the Department Manager, Project Manager, and QA Officer may be needed to determine the proper corrective action. After review and acceptance, the file is submitted to the Group Leader/Department Manager for secondary review.

12.1.4 - The Group Leader/Department Manager provides secondary review of the analytical data. This includes review of any associated quality control results. The review also includes a spot check of the summarized analytical data to ensure that the data sheets reflect the values in the raw data package. Any discrepancies are corrected, initialed, and dated. After review and acceptance, the file is submitted to the Project Manager for final review.

12.1.5 - The Project Manager, who is knowledgeable about the site or process generating the sample, provides the final data review. This review consists of comparison with historical data, a cross-check of client sample information, and a final assessment that standardized data reporting conventions have been employed.

12.1.6 - The Project Manager submits the data to the client with the potential existing for further review by the client's QA personnel.

12.2 Documentation Checks

During data reduction and assembly, several documentation checks and data transfer steps are performed. The following is a list of the specific checks performed during data reduction and assembly.

12.2.1 Laboratory Notebooks

Laboratory notebooks are maintained by analysts and periodically reviewed by the laboratory supervisor to ensure that they are kept up to date and that the procedures followed are consistent with standard operating procedures. In addition, photocopies of the laboratory notebooks may be produced and submitted with the raw analytical data package.

12.2.2 Laboratory Benchsheet

Analysts employ a number of laboratory benchsheets to record calculations, QC results, calibration curve results, and other applicable data generation mechanisms. All original laboratory benchsheets are included in the raw data packages for review by data review personnel. Laboratory benchsheets show the worksheet description clearly printed across the top of the document.

12.2.3 Checks on Raw Data Calculations

Data management review staff routinely perform checks on the raw data calculations to ensure that the analyst is performing the calculations properly. This form of data review is very effective for identifying systematic errors and can also assist in identifying random errors.

12.2.4 Checks on Direct Computerized Data Transfer

To facilitate proper checks on instrumental data which has been electronically transferred to a computer text, spreadsheet, or ASCII file, data review is performed prior to that transfer. In addition, software programs originating at ENCOTEC are reviewed to ensure that the raw data is handled using proper formulae for calculations, significant figures, integration, etc.

12.2.5 Checks on Raw Data Packages

Each laboratory within ENCOTEC has specific raw data package organization requirements that are needed to standardize the data review process. As the data packages are assembled by the analyst, a check off system is used to catalogue the data package elements. All raw data packages receive primary and secondary reviews.

12.2.6 Analytical Logs

A number of analytical logsheets are employed by ENCOTEC (such as sample preparation logs, instrument run logs, analytical procedure logs, etc.) to enable the analyst to

record the preparation and analysis of samples. Most original laboratory logsheets are included in the raw analytical data packages for review by data review personnel.

12.2.7 Calibration Summary Logsheets

ENCOTEC routinely performs a variety of calibrations prior to sample analysis. It becomes paramount that calibration data be scrutinized to ensure that the values reported are valid. Verification of the curve is generally performed at the analyst level and is validated by a secondary review.

12.2.8 Internal Chain of Custody

The internal chain-of-custody system has proven to be an effective way to document custody transfers within the facility. These custody documents remain a part of the ENCOTEC document files. Intralaboratory custody is maintained by a sample librarian and the documents are periodically reviewed by the Laboratory Support Group Leader.

12.2.9 QC Acceptance Criteria

ENCOTEC-established or reference method QC acceptance criteria are noted on laboratory bench sheets and are available to the analyst at all times. It is the responsibility of the analyst to ensure that the established criteria are met. If they are not, the non-compliance should be documented, the proper supervisory personnel notified, and appropriate corrective action taken. The situation may

ultimately be brought to the attention of the Department Manager, Project Manager, QA Officer, and/or Technical Director. Any additional corrective action will be performed at the direction of these individuals.

12.2.10 Summarized Data Entry

Summarized data entry is performed after the raw data has been reviewed and accepted at the secondary review stage. It is then entered into a computer for final report production. The summarized data is then proofread for correctness by the Project Manager or quality assurance staff.

12.3 Sample and Data Identification

ENCOTEC utilizes a number of labeling mechanisms to ensure that raw data accurately identifies the samples received for analysis. The identifier used for all analytical data generated for a sample is the ENCOTEC ID. This number is used to identify samples on all chromatograms, bench sheets, instrument run logs, laboratory worksheets, and chain-of-custody forms. Other information common to data production and review documents includes date of analysis, analysis parameters, analyst performing the analysis, project name, project number, and all intermediate values used in the data calculations. In addition, data production and review documents such as chromatograms or other raw instrument outputs contain additional information such as time of analysis, retention times, instrument conditions and integration methods.

12.4 Logbooks

On a regular basis, each analyst maintains the log books associated with sample preparation and/or analysis. Log books, notebooks or file logs are organized on a functional basis. The following logs are used:

- Sample Extraction and Cleanup Log (one per prep area)
- GC/HPLC Instrument Log
- GC/HPLC Maintenance Log
- GC/HPLC Magnetic Tape Log
- GC/HPLC Reference Standard Preparation Log Book
- GC/MS Reference Standard Preparation Log
- GC/MS Instrument Log
- GC/MS Maintenance Log
- GC/MS Magnetic Tape Log
- Inorganic Sample Preparation Log Book (one per prep area)
- Inorganics Reference Standard Preparation Log Book
- AA Maintenance Log (one per instrument)
- ICP Maintenance Log
- ICP\MS Maintenance Log
- TRAACS Maintenance Log
- OI 700 TOC Maintenance Log
- LACHAT Maintenance Log
- IC Maintenance Log
- Balance Calibration Log Book
- Eppendorf Calibration Log Book
- pH Calibration Log

All log sheets must be dated and signed by the analyst making the entries.

12.4.1 Organic Sample Extraction and Cleanup Log Book

At the beginning of each period, a new page in the sample extraction and cleanup log book is started. The data is entered on the sheet and sample preparation begins. All appropriate information and comments must be filled in for each sample extracted or prepared. The log sheet is set up for entry in chronological order and samples should be entered in the order of handling. At the end of the day after samples have been returned to the sample storage area, the analyst must check for completeness of the log form. Pertinent chain-of-custody information, surrogate/matrix spike identification, and balance checks (soil extractions) performed must be entered. Additional chain-of-custody tracking is entered on the organics cleanup form.

12.4.2 GC Instrument Log

A log of instrument conditions and samples analyzed is maintained by the sequence tables from the computer runs and from the associated "method" defined for the computer-acquired data. The information provided by these sequence tables is divided into two types. The first set of information is the method being used for acquisition (determined by the analyst) include operating information on column type, temperature conditions, detector, etc. This information is printed on the header of each sample report.

The second set of data includes information on the samples, standards, and blanks run on the instrument. The sequence table must be built before each set of analyses are performed. This table is the reference used to generate the

header information on each sample. This sequence table will then be entered in the run log file for future reference. These run logs provide a history of instrument use.

12.4.3 GC Maintenance Log

A GC maintenance log is employed to record maintenance and any special calibrations or electronic checks performed on the unit. This notebook is to be used anytime a unit is down for maintenance, column changing, detector cleaning, etc.; it records time and date of equipment service and a description of the problem encountered and repairs accomplished. Each page should be signed by the person performing the work or assigned to work with an outside repair technician.

12.4.4 GC/HPLC Magnetic Tape Log

All data generated by GC is transferred for long term storage to magnetic tape. A log book is maintained to document the tape location of the archived data and to permit retrieval of this data at a later date if requested.

12.4.5 GC/HPLC Reference Standard Preparation Log Book

A reference standard preparation log book is maintained in order to reference each calibration mixture, matrix spike, or surrogate compound which is either made from neat compounds or diluted from a stock solution.

An entry is made in this log book each time a new reference mixture is prepared. Each entry will contain the date, and name of analyst making the mixture. If this reference solution is being prepared from EPA-referenced stock solutions the following information is entered: name of compound, concentration of stock, volume of stock used to prepare dilution, final volume of dilution, concentration of compound in dilution, and solvent used for dilution. The new working standard is given a standard reference number which is logged in the book and on the vial.

If the reference mixture is made from neat compounds, the tare weight, final weight, and net weight are recorded in the log book as well as all information described above.

12.4.6 GC/MS Reference Standard Preparation Lab Book

A reference standard preparation log book is maintained in order to reference each calibration mixture, internal standard, or surrogate compound which is either made from neat compounds or diluted from a stock solution.

An entry is made in this log book each time a new reference mixture is prepared. Each entry will contain the date and name of the analyst making the mixture. If this reference solution is being prepared from EPA-referenced stock solutions the following information is entered: name of compound, concentration of stock, volume of stock used to prepare dilution, final volume of dilution, concentration of compound in dilution, and solvent used for dilution. The new working standard is given a standard reference number which is logged in the book and on the vial.

If the reference mixture is made from neat compounds, the tare weight, final weight, and net weight are recorded in the log book as well as all information described above.

12.4.7 GC/MS Instrument Log

An instrument log is located next to the GC/MS for recording work performed each day on the instrument. This log is to be used to record basic information on instrument conditions during sample runs and to maintain a chronological list of every sample run made each day.

Each run performed on the GC/MS is recorded on this log sheet. All information should be completed by the analyst. The log sheet consists of an original plus a carbon copy. The original sheet is to pulled from the book and submitted with the chromatograms, quantification reports and all other data output from the computerized system. This data sheet along with the computer reports will be used to verify calculations and machine conditions. Ultimately, the log is placed in the document control central files. The carbon copy remains in the notebook for future reference. This copy shows a complete history of the machine operations during any given period of time.

In addition to the manual log, the computerized output for each sample run contains information for that particular run. The following information is to be entered at the terminal for each run made:

- Sample Number
- Project Number
- Case Number
- Instruments
- Type of Analysis (VOA, BNA, etc.)
- Multiplier Setting
- Volume Injected
- Column Used
- Purge Rate (if applicable)
- Temp Prog Rate (if applicable)
- Split or Splitless

This information is printed by the computer system for each analysis and serves as the data identifier.

12.4.8 GC/MS Maintenance Log

A second notebook is maintained with the GC/MS unit to record all maintenance or service performed on the unit. Each time the instrument is down for maintenance, entries are made in this log. Types of information to be included are ion source cleaning, parts replacement, new GC column installation, etc. Any activity relating to the operation of the unit must be recorded. A new page is started for each new day and signed by the analyst who performs the work. This maintenance log represents a history of the instrument itself, problems/solutions and any other information on machine conditions.

12.4.9 GC/MS Magnetic Tape Log

In order to facilitate retrieval of stored data, an inventory or log of GC/MS data files is maintained for each magnetic tape. An example of this tape log follows. Each log contains the data file name, case number, date, and length of file.

12.4.10 Inorganic Sample Preparation Log Book

Sample preparation log books are located at each preparation station. At the beginning of each day or set, a new page in the book is started. The data is entered on the sheet and sample preparation begins. All appropriate information and comments must be filled in for each sample extracted or prepared. The log sheet is set up for entry in chronological order and samples should be entered in order of handling. At the end of the day, after samples have been returned to the sample storage area, the analyst must check for completeness of the log form. Once the accuracy of the information on the form is verified, the form should be initialed.

12.4.11 Inorganics Reference Standard Preparation Log Book

A reference standard preparation log book is maintained in order to reference each calibration mixture which is either made from neat compounds or diluted from a stock solution.

An entry is made in this log book each time a new reference mixture is prepared. Each entry will contain the date and name of analyst making the mixture. If this reference solution is being prepared from EPA referenced stock solutions, the following information is entered: name of compound, concentration of stock, volume of stock used to prepare the dilution, final volume of the dilution, concentration of compound in the dilution, and solvent used for dilution.

If the reference mixture is made from neat compounds, the tare weight, final weight, and net weight are recorded in the log book as well as all information described above.

12.4.12 AA Maintenance Log

An AA maintenance log is maintained for each AA unit to record maintenance and any special calibrations or electronic checks performed on the unit. This notebook is to be used anytime the unit is down for maintenance. This book is used to record time and date of equipment service, a description of the problem encountered, and repairs accomplished. Each page should be signed by the person performing the work or assigned to work with an outside repair technician. These forms will be reviewed by the Group Leader periodically.

12.4.13 ICP Maintenance Log

A notebook is maintained with the ICP unit to record all maintenance or service performed on the unit. Each time the instrument is down for maintenance, entries are made in the log. Types of information to be included are parts cleaning,

parts replacement, etc. Any activity relating to the operation of the unit must be recorded. A new page is started for each new day and signed by the analyst who performs the work. This maintenance log represents a history of the instrument itself, problems/solutions, and any other information on machine condition.

12.4.14 ICP\MS Maintenance Log

A notebook is maintained with the ICP/MS unit to record all maintenance or service performed on the unit. Each time the instrument is down for maintenance, entries are made in the log. Types of information to be included are parts cleaning, parts replacement, etc. Any activity relating to the operation of the unit must be recorded. This maintenance log represents a history of the instrument itself, problems/solutions, and any other information on machine condition.

12.4.15 TRAACS, OI 700 TOC, LACHAT and IC Maintenance Logs

These logs are used whenever routine maintenance is performed on the instruments as well as whenever manufacturer service or preventive maintenance is performed. These notebooks provide entries for date, type of maintenance procedure, repair parts ordered, and analyst's initials.

12.4.16 Balance Calibration Log Book

This log serves to monitor reliability and accuracy of weight measurements. The notebook provides log sheets with entries for the date, transcription of the balance read-outs for the three class "S" weight used, the analyst's initials and supervisor's signature.

12.4.17 Eppendorf Calibration Log Book

This notebook provides log sheets with entries for the date, analyst's initials, Eppendorf ID number, designated volume (mL), weights and average weight for three aliquots, and the percent accuracy.

12.4.18 pH Calibration Log

Provisions are made in this notebook for recording the meter number, date, readings and % accuracies for two buffer buffers, the slope and the analyst's initials.

12.5 Analytical Data Validation

ENCOTEC is capable of performing data validation in order to ensure the integrity of the reported data. Data validation, however, is a term that carries several connotations. ENCOTEC has classified data validation in the following ways:

12.5.1 Internal Data Validation - This type of validation is performed on selected analytical data. Laboratory narratives, if requested by the client, are also produced with final approval by the QA Officer or designate.

12.5.2 Internal Data Validation by Agency/Client Specific Procedures - ENCOTEC can perform internal data validation using procedures specified by the client or a particular agency. These data validation guidelines are based on client-specific SOP's or any of the following documents:

a) Laboratory Data Validation Functional Guidelines for Evaluating Organics Analysis. Prepared for the Hazardous Site Evaluation Division of the U.S. Environmental Protection Agency, February, 1988.

b) Region II Standard Operating Procedure for the Evaluation of Metals Data for the Contract Laboratory Program. U.S. Environmental Protection Agency, February, 1990.

c) Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analysis. Prepared for the Hazardous Site Evaluation Division of the U. S. Environmental Protection Agency, October, 1989.

12.5.3 External Data Validation by Agency/Client Specific Procedures - ENCOTEC maintains the ability to produce analytical data packages for external (outside, independent or ENCOTEC-contractor) data validation by any of the above data validation procedures.

12.6 Data Generation and Handling Events

All analysts record results and QC data on pre-printed forms. These are checked by data review personnel, group leaders and quality assurance staff for correctness in calculations, units, significant figures, QC calculations and instrument calibrations. The summarized data is transmitted by hardcopy to the project manager. Project managers guide the handwritten forms through the clerical department. Typists retrieve the appropriate report form from the local area network (LAN) using a form code unique to the document. The third level of verification occurs when project managers check the word processor version against the handwritten version for transcription errors. The completed forms are then photocopied and collated into reports.

Cover pages and narratives are generated when requested by contract and can be provided according to client specifications. Narratives always cover any quality control problems and corrective actions, such as reanalysis, any difficulties in sample preparation and analysis, such as matrix interferences, and apparent sampling problems. Narratives from each work group are electronically mailed over the LAN to a QA staff member for review and approval.

12.7 Data Reporting

12.7.1 Minimum Data Reporting Requirements

Analytical data can be presented in a number of formats. These formats are generally based on either the needs of the client or on the Data Quality Objectives specified for the project. In general, most analytical reports contain at a minimum the following information:

- Client Name
- Project Number
- Report Date
- Analysis Method Number
- Client Sample Identification
- ENCOTEC Sample ID
- Date of Sample Collection
- Date of Sample Receipt
- Date Sample was Prepared
- Date of Sample Analysis
- Analytical Parameters
- Analytical Detection Limits
- Analytes Detected
- Report-specific Data Flags

12.7.2 Reporting Conventions

Reporting conventions have been established to ensure that analytical data and quality control results are entered in a consistent and correct manner, regardless of the laboratory/department of origin.

ENCOTEC uses a variety of flags to qualify reported analytical data (see ENCOTEC SOPs for data reduction and reporting conventions).

U Flag - This flag is used in cases when the analyte is not detected at or above the specified detection limit.

J Flag - This flag is used in cases when the analyte is detectable but cannot be quantified (within standard precision and accuracy guidelines).

B Flag - This flag is used in cases when the analyte is present in the laboratory method blank.

E Flag - This flag is used in cases when the value is estimated.

S Flag - This flag is used to identify results from a (secondary) dilution.

12.7.3 Calibrations

12.7.3.1 Temperature Compensation - Temperature compensation is required by several analytical methods due to the bias effects of temperature on the analysis. Examples include pH meters and conductivity meters.

12.7.3.2 Cell Constants - Cell constants are required for several spectrophotometric analyses. Analytical precision is increased by performing the analysis of samples and analytical standards using the same cells.

12.7.3.3 Regression Analysis - Least squares linear regression is utilized for many standard calibrations. Linear regressions are, in the majority of cases, performed at the instrument level with correlation coefficients and/or percent relative standard deviation

(%RSD) evaluated to determine the acceptability of the curve.

12.7.3.4 Internal Standard Quantification - Several analytical methods require internal standard quantification techniques. This technique is used in all procedures utilizing mass spectrometry. All mass spectrometers are interfaced to computers which utilize vendor-supplied software which calculates the Minimum Relative Response Factors (RRF) and Extracted Ion Current Profile (EICP) data used to determine acceptance of calibration curves. (EICP data are also monitored for sample analyses.)

12.7.4 Reporting Formats

In general the report formats produced by ENCOTEC are based on the method and parameter list. Depending upon the project, deliverable reports (QC Levels 0 - 4) can be produced. They can be as simple as consisting of the summarized data results or as complex as a full USEPA Contract Laboratory Program Deliverables Package with data validation.

Forms are produced in accordance with ENCOTEC data reporting conventions and client specifications. They are created by the Technical Documentation Officer subject to management staff approval required prior to implementation. A spreadsheet forms management system facilitates systematic revision of forms and purging of unused forms. All forms contain identifying codes to ensure that transcriptions involve the correct forms. New report forms are routinely

created for clients according to their specifications and, because the forms are collated manually, report organization can also be in any manner the client desires.

12.8 Records Management

A system is in place at ENCOTEC to ensure that records are maintained in good condition and are retrievable. An effective safeguard is thereby provided to ensure that data records generated by ENCOTEC are preserved in a consistent manner.

12.8.1 Generation of Records

All ENCOTEC records are required to be legible, accurate, complete and are to be archived in such a way that they be easily reproduced. All original hardcopy results should be written using dark indelible ink. All computer media records should be labeled with references as to the content of the tape/diskette, or contain a hardcopy record which defines the operating system, programs listing and file names.

12.8.2 Acceptance of Records

All records which are submitted to the ENCOTEC archives should be complete and final.

12.8.3 Preservation of Records

Records will be stored to preclude avoidable deterioration or loss. The following steps are taken to ensure that records are preserved for as long as possible:

- Temperature and humidity are controlled.
- Records will be stored in steel filing cabinets or on steel shelving.
- Provisions are made for records such as photographs and negatives so as to prevent damage from excessive light, stacking, etc
- Computer media will be stored in a write-protect (read only) mode.

12.8.4 Safekeeping of Records

Access to the records storage area will be limited to those employees within ENCOTEC who have a clear and intended purpose requiring access. All access by non-ENCOTEC personnel will be granted only by management consent, conditioned on a escorted tour. Eating, drinking or the use of tobacco products is prohibited in the records storage area. ENCOTEC personnel will periodically police the records area to ensure that the records are stored in a manner consistent with the policies of this section.

12.8.5 Corrections to Records

In the unlikely event of records correction, the corrections will be noted, initialed (or signed) and dated. The corrections should be done in such a way as not to obliterate the incorrect entry. The use of correction fluid in the correction of laboratory bench data is not permitted.

12.8.6 Retrieval of Records

Provisions are made to ensure that records can be quickly and effectively retrieved within the planned retention period.

12.8.7 Retention of Records

All data records will be retained by ENCOTEC for a period of not longer than seven years after transmittal (unless special arrangements are made by the client).

13.0 Corrective Action

13.1 Introduction

Analytical measurements must conform to established quality control guidelines for the particular method in question. When deficiencies and nonconformance to established guidelines are present, corrective action is necessary to bring the analytical process back into control. The initial objective of any corrective action is the identification of the problem and the determination as to whether the out-of-control event is transient in nature or a systematic phenomenon. Additionally, corrective action involves investigation into resolution of the problem and implementation of the solution. An effective corrective action includes monitoring the situation after a solution has been implemented to ensure that the solution remains effective.

Examples of corrective actions are:

- sample reanalysis
- sample re-extraction/redigestion
- standard recalibration
- instrument maintenance
- resampling
- additional training/instruction

13.2 QA Officer/Management Responsibility

The Quality Assurance Officer has responsibility for overseeing the corrective action process. Improvements and upgrades to the corrective action SOP will be recommended by the QA Officer after review with senior management. Additionally, the QA

Officer is involved with specifying corrective actions for identified deficiencies/nonconformities and monitoring the implementation and success of those actions.

13.3 Documentation

All significant deficiencies/nonconformities are documented and are maintained in formats such as:

- part of a data package
- laboratory logbook
- request for sample re-extraction
- formal request for a corrective action response for (any) deficiencies noted following a laboratory audit or performance evaluation
- service/repair records
- laboratory narrative (upon request)

For example, laboratory narrative sections of analytical reports will contain descriptions of any corrective actions required and are submitted as part of the analytical report. Other corrective action documentation will be kept on file and available to clients upon request.

14.0 Quality Assurance Audits

Quality assurance audits are comprised of external and internal audits. Reports are issued to management regarding the audits.

14.1 External Audits

14.1.1 Federal agency audits

ENCOTEC participates in federal, state, and private industry programs for both organic and inorganic methods evaluation, laboratory evaluation, and assessment of analytical capabilities. Under the USEPA Contract Laboratory Program, performance evaluations are performed on blind check samples in addition to onsite laboratory audits to ensure compliance with contract Statements of Work (SOW). These audits occur, at a minimum, annually. ENCOTEC also participates in both the Water Pollution (WP) and Water Supply (WS) Performance Evaluations administered by the EPA. In addition to the above, comprehensive lab audits have been performed by EPA Region V and the USEPA National Enforcement and Investigation Center (NEIC) both for pre-contract award and for compliance in conjunction with analytical services rendered to clients.

A list of the various audit programs and performance evaluations in which ENCOTEC participates can be found in Table 14.1.

14.1.2 State agency audits

ENCOTEC is also audited by the State of New Jersey's Department of Environmental Protection and Energy under its Laboratory Certification program, along with the analysis of blind check samples during performance evaluations. Successful continued performance on semi-annual WP performance evaluations is a requirement for continued certification. These audits are conducted on a semi-annual basis. The State of Michigan currently does not have a certification/lab audit program for non-potable water and/or solid waste.

14.1.3 Private client audits

ENCOTEC is also audited by some of its clients from the private sector. Annual/semi-annual laboratory audits as well as blind and double blind spike samples may be included in these programs.

14.2 Internal Audits

Internal audits of the entire ENCOTEC System are conducted periodically. These evaluations consist of a review of the operations of each laboratory or department in the context of the potential impact on the quality and defensibility of the data produced. The major areas that may be addressed include:

- Sample Receipt
- Chain of Custody
- Sample Preparation
- Sample Analysis
- Quality Control Analysis Frequency
- Standard Traceability
- Performance Evaluation results
- Corrective Action
- Data Reduction
- Data Review and Approval
- Documentation

Audits are conducted by the following senior staff:

- Quality Assurance Officer and/or Quality Assurance Chemist
- Laboratory Manager, Technical Director, and/or Department Manager depending upon the scope of the audit

Staff contacted include:

- Group Leaders
- Supervisors
- Senior Chemists
- All Chemists and Technicians

Monthly audits are conducted for the purpose of log book maintenance in the General Chemistry Laboratory, Metals Laboratory, and Waste Screens Laboratory. The items routinely inspected are:

- All instrument maintenance log books.
- Standards Preparation and Traceability Log Book
- Balance Calibration Log Book
- Eppendorf Calibration Log Book
- Prepared or purchased standards Logs
- Sample Support Group Radiation Check Log

Instrument maintenance logbooks are inspected to ensure that entries are current, legible and complete.

Standards logbooks are inspected to make sure that purchased and prepared standards are traceable and that preparations are documented. The standards are checked to ensure that required information is present to correctly identify the standard and its source, that only current standards are used and that expired ones are removed from use.

The calibration logbooks provide evidence that calibrations are carried out at the specified frequencies.

A regularly monitored item is the Radiation Check Log. All incoming samples are routinely monitored with a survey meter for alpha, beta or gamma radiation. This is provided to ensure that personnel are not unduly subjected to radiation, and to remain compliant with OSHA regulations.

14.3 Quality Assurance Reports

14.3.1 Internal Reports

Internal Reports which summarize the performance of laboratories on EPA (or other agency/private client) Performance Evaluations are provided to management upon receipt of results and occur approximately eight times per year. Included are Water Supply (WS), Water Pollution (WP) and Contract Laboratory Program (CLP) results. Internal memos are also provided in response to laboratory audit findings.

Reports are prepared by the Quality Assurance Officer and are distributed to the following personnel:

- President
- Executive Vice-President
- Vice-President
- Technical Director, Laboratory Manager, Department Managers
- Group Leaders

14.3.2 External Reports

External Reports are provided to clients and/or regulatory agencies depending upon the scope of the project and the nature of the response needed.

Laboratory narratives which provide a text summary of the important aspects of the analysis of a group of samples and include an assessment of the quality control results and the need (if any) for corrective action can be generated. These narratives are transmitted to the client based upon the unique reporting needs of the project.

If requested, and based upon the needs of the project, a more extensive summary detailing the quality control results, corrective action (if needed), and other information of specific interest, can be generated. This type of report is also a summary of the individual laboratory narratives generated over the duration of the project and will be more extensive than individual laboratory narrative reports.

TABLE 14.1

Agency/Client	Program	Audit	Evaluations
USEPA	Contract Lab	Annual	Quarterly Program (CLP)
USEPA, Region V	Water Pollution	----	Semi-annual
USEPA, Region V	Water Supply	----	Semi-annual
State of New Jersey, Department of Environmental Quality and Energy	Laboratory Certification	Biennial	Initial, every 2 years thereafter
Private Sector	Client Specific	Annual	Variable

15.0 Conclusion

The commitment to quality assurance at ENCOTEC is firm and unyielding. However, the QA program designed to fulfill this commitment must be dynamic and flexible in order to keep up with the needs of new methodologies, new instrumentation, new theories of practice, and new regulations.

Therefore, scheduled reviews of the QC program are performed to ensure that the program remains current and relevant to the needs of the Chemistry Section, the company, and our clients. Staff meetings are held to discuss various aspects of the QC program, allowing analysts to become more involved in planning and trouble-shooting and to become active participants in improving the overall quality of data generated by the Chemistry Section. The goal of these meetings is to:

1. Identify problem areas and recommend improvements or alterations in the QA program.

2. Seek, evaluate and discuss new ideas and current developments in the field of QA and recommend means for their application.

3. Inform the section and affected project managers of specific instances of non-conformance to the QA program.

Any changes in the QA program are always given final approval by the Technical Director, Quality Assurance Officer and senior management of the company before they become effective.

In conclusion, the ENCOTEC chemistry section quality control mechanisms, quality assurance management, and constant evaluation and review of the QA program, as a whole, enable the Section to fulfill its primary purpose. The purpose and goal of the Section is to produce and report data that is scientifically valid and defensible.

REFERENCES

1) Handbook for Analytical Quality Control in Water and Wastewater Laboratories; U.S. Environmental Protection Agency. Environmental Monitoring and Support Laboratory, Cincinnati, OH, 1979; EPA 600/4-79-019.

2) Test Methods For Evaluating Solid Waste - Quality Control; Third Edition; Environmentat Protection Agency. Office of Solid Waste and Emergency Response, Washington, DC, 1986; EPA SW-846.

3) Carson, Williams A Guide to Quality Control Practices For Waste and Potable Water Analysts; Fourth Edition; Environmental Resource Associates, Arvada, Colorado, 1988.

4) Taylor, John Keenan, Quality Assurance of Chemical Measurements; Lewis, Chelsea, Michigan, 1987.

5) U.S. EPA Contract Laboratory Program - Statement of Work For Organics Analysis - Multi-media, Multi-concentration; U.S. Environmental Protection Agency. Analytical Operations Branch. Superfund Contract Office, Washington, DC.

6) NIOSH Manual of Analytical Methods; Volume Three, Second Edition; U.S. Government Printing Office, Washington, DC, 1977; GPO No. D17-033-00261-4.

7) Compendium of Methods For the Determination of Toxic Organic Compounds in Ambient Air; U.S. Environmental Protection Agency. Environmental Monitoring Systems Laboratory - RTP, Research Triangle Park, NC, 1986; EPA 600/4-87-006.

Appendix

A) Instrumentation and Equipment List

B) Data Organization and Review Forms

- Inorganics Department
- GC/MS
- GC/HPLC and Organic Extractions

Appendix A

MAJOR ANALYTICAL INSTRUMENTATION

Instrumentation Description	Manufacturer	Model	Serial Number	Installation Date
ENCOTEC I (3985 Research Park Dr.)				
GC/HPLC Laboratory				
Automated Volatiles Autosampler	Tekmar	ALS 2016	91344009	1992
Automated Volatiles Autosampler	Tekmar	ALS 2016	92197001	1992
Automated Volatiles Concentrator, Purge and Trap	Tekmar	LCS 2000	92232009	1992
Automated Volatiles Concentrator, Purge and Trap	Tekmar	LCS 2000	92120007	1992
Automated Volatiles Concentrator, Purge and Trap	Tekmar	LCS-2000	90362011	1991
Automated Volatiles Concentrator, Purge and Trap	Tekmar	ALS-2016	90344014	1991
Automatic Sample Heater	Tekmar	Not Available	92009002	1992
Automatic Sample Heater	Tekmar	Not Available	92266007	1992
Automatic Sample Heater	Tekmar	Not Available	90347007	1991
FID Electrometer	GOW MAL	40-900	F68810	1992
GC Autosampler	Varian	8200	5337	1992
GC Autosampler	Varian	8200	5338	1992
GC Autosampler	Varian	8200	5155	1992
GC Autosampler	Varian	8100	2531	1991
GC Autosampler	Varian	8100	2132	1991
GC Autosampler	Varian	8100	0603	1990
GC Autosampler	Varian	8100	0604	1989
GC Autosampler	Varian	8035	Z9JA88	1987
GC Autosampler	Varian	8034	41387N086	1986
GC Autosampler	Varian	8034	3524	1986
GC Autosampler	Varian	8034	3824	1986
Gas Chromatograph, Dual ECD	Varian	3400	15491	1991
Gas Chromatograph, Dual ECD	Varian	3600	0407	1989
Gas Chromatograph, Dual ECD	Varian	3600	0617	1989
Gas Chromatograph, Dual ECD	Varian	6000	3524	1986
Gas Chromatograph, Dual ECD	Varian	3700	45519130-13	1979
Gas Chromatograph, Dual ECD/FID	Varian	Star 3600	1388	1992
Gas Chromatograph, Dual TSD	Varian	3400	8239	1990
Gas Chromatograph, ECD/FID	Varian	3400	0787	1984
Gas Chromatograph, FID/NPD	Varian	3700	73130518-13	1986
Gas Chromatograph, HECD/PID	Varian	3600	01377	1992
Gas Chromatograph, HECD/PID	Tracor	540	871222	1987
Gas Chromatograph, FID/TSD	Varian	3400	14455	1991

MAJOR ANALYTICAL INSTRUMENTATION (con't.)

Instrumentation Description	Manufacturer	Model	Serial Number	Installation Date
ENCOTEC I				
GC/HPLC Laboratory (con't.)				
Gas Chromatograph, HECD/PID/FID	Varian	3400	4729	1989
HPLC Fluorescence Detector	Waters	470	001723	1992
HPLC W/UV Detector	Waters	LC Module 1	LCM100260	1992
GC/MS Laboratory				
Automated Volatiles Concentrator, Air Canister Sampler	ENTECH	M2000	00007	1991
Automated Volatiles Concentrator, Purge and Trap	Tekmar	ALS - 2016	91149004	1990
Automated Volatiles Concentrator, Purge and Trap	Tekmar	LCS - 2000	91141010	1990
Automated Volatiles Concentrator, Purge and Trap	Tekmar	LCS - 2000	90254005	1990
Automated Volatiles Concentrator, Purge and Trap	Tekmar	LCS - 2000	88166001	1989
Automated Volatiles Concentrator, Purge and Trap	Tekmar	LCS - 2000	88181005	1989
Automated Volatiles Concentrator, Purge and Trap	Tekmar	ALS - 2016	89116016	1989
Automated Volatiles Concentrator, Purge and Trap	Tekmar	ALS - 2016	90249011	1989
GC Autosampler	Varian	8034	89115002	1989
GC Autosampler	Hewlett-Packard	18594A	Not Available	1987
GC Autosampler	Hewlett-Packard	18594A	3113A26632	1991
GC Autosampler	Varian	8034	3113A26444	1991
GC Autosampler	Varian	8100	076824	1987
GC Autosampler	Varian	8034	2387	1992
Gas Chromatograph/ Mass Spectrometer, Ion Trap	Varian	Saturn (ITD 40)	076971	1989
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Finnigan	4500	IS 003165	1991
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Finnigan	Incos 50	1210305-0680	1981
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Finnigan	3200	IN0323	1989
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Finnigan	5100-SP	11536-0679	1980
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Finnigan	Incos 50	14275-0884	1987
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Finnigan	5100-SP	IN0456	1989
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Hewlett-Packard	5971A	14305-0887	1989
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Hewlett-Packard	5971A	3118A02685	1991
Gas Chromatograph/ Mass Spectrometer, Quadropole	Hewlett-Packard	5971A	3118A02684	1991
Thermal Desorption Unit	Tekmar	5010	3188A03514	1992
Thermal Trap Conditioner	Tekmar	5100	89054015	1990
			90008001	1990

MAJOR ANALYTICAL INSTRUMENTATION (con't.)

Instrumentation Description	Manufacturer	Model	Serial Number	Installation Date
ENCOTEC I				
Organic Extractions Laboratory				
Analytical Balance	Satorius	B31005-OUR	40010029	1990
Analytical Balance	American Scientific	DTL500S	19530	1987
Auto Concentrator	ABC Laboratories	601-602	601027-043-024	1989
Benchmate Workstation	Zymark	BFS1	04453	1992
Benchmate Workstation Collector (X-Y)	ISCO	Foxy 200	622130050-92177	1992
Continuous Liquid-Liquid Extractors (Twelve)	Corning	3928-M	Not Available	1991
Drying Oven	Equatherm	Not Available	905661953	1990
GC Autosampler	Varian	8100	2530	1992
GC Autosampler	Varian	8034	37224MY85	1986
GC Autosampler	Varian	8034	3864	1985
Gas Chromatograph, ECD	Varian	Star 3400	15490	1992
Gas Chromatograph, ECD	Varian	3400	3006	1986
Gas Chromatograph, FID	Varian	3700-2	73130518-13	1985
Gel Permeation Chromatograph	ABC Laboratories	1002	592A	1987
Hot Plate Stirrer	Corning	PC-351	Not Available	1989
LC Pump	Sci. Systems, Inc.	30016-0178	E2907439	1992
Muffle Furnace	Thermolyne	30400	54400442	1990
N-Evap Evaporator	Organomation	111	6788	1990
N-Evap Evaporator	Organomation	111	4033	1987
Recorder	Esterlene Angus	Not Available	S-2243-18	1986
Separatory Funnel Shaker	Eberbach	Not Available	Not Available	1990
Separatory Funnel Shaker	Eberbach	Not Available	Not Available	1990
Soxlets (ten)	Kimax	Not Available	Not Available	1986
Steam Bath	Boekel	Not Available	041089-01	1989
Steam Bath	Boekel	Not Available	08286-09	1986
Steam Bath	Precision Scientific	Not Available	11AX-4	1990
TOX Analyzer	Cosa	tox10	75C00822, 75A00070, 75P10252	1992
TOX Analyzer	Cosa	tox10	43D30438, 43R30483, 43C0438	1988
TOX Sample Preparator	MCI	TOX-10-A	43A31272	1992
TOX Sample Preparator	MCI	TOX-10-3	43A30208	1988
Tube Heater	Kontes	72001-0000	1689	1990
Tube Heater	Kontes	72001-0000	1661	1990
Tube Heater	Kontes	72001-0000	1694	1990

MAJOR ANALYTICAL INSTRUMENTATION (con't.)

Instrumentation Description	Manufacturer	Model	Serial Number	Installation Date
ENCOTEC I				
Organic Extractions Laboratory (con't.)				
Tube Heater	Kontes	72001-0000	1564	1987
TurboVap Evaporator	Zymark	TurboVap	4329	1990
UV Detector	ISCO	Typell 92076	182946	1992
UV Detector	Milton Roy	Not Available	036919	1987
Ultrasonic Disruptor	Heat Systems	XL2020	G1024	1990
Ultrasonic Disruptor	Heat Systems	XL2020	G1025	1990
Ultrasonic Disruptor	Heat Systems	W-385	G8847	1989
Ultrasonic Disruptor	Heat Systems	W-375	G7042	1987
Vacuum Manifolds (three)	Supelco	Not Available	Not Available	1989
Vacuum Oven	Baxter	DP-32	192003	1990
Vapor Recovery System	Glas-Col	CX50212	240393	1992
Vapor Recovery System Control Box	Glas-Col	PL3126	257097	1992
Water Recirculator	Lauda	MS 3	N08010	1991
Water Recirculator	Lauda	UKT 1500 P	M05015	1991
Water Recirculator	Lauda	MS 3	N08015	1991
Water Recirculator	Lauda	MS 3	N08013	1991
Water Recirculator	Lauda	MS 3	N08017	1991
pH Meter	Corning	240	5635	1989
Metals Laboratory				
AA Autosampler	Varian	PSC - 56	1051037	1991
AA Autosampler	Varian	PSC - 56	8081211	1991
AA Autosampler/Furnace	Perkin- Elmer	AA - 70	193978-5807	1991
Atomic Absorbtion Unit, Flame/Furnace	Varian	AA20	0011263	1986
Atomic Absorption Unit, Flame	Varian	AA20 ABQ	612 1269	1988
Atomic Absorption Unit, Furnace GTA	Varian	GTA-96	606 1312	1986
Atomic Absorption Unit, Furnace w/Zeeman Background Correction	Perkin- Elmer	4100ZL	6059	1991
Cold Vapor Generator	Varian	VGA - 76	9101295	1990
Cold Vapor Generator	Varian	VGA - 76	509 0389	1988
ICP Autosampler	ARL	AIM - 101	161-521	1989
ICP Autosampler	Perkin- Elmer	AS - 50	133080	1987

MAJOR ANALYTICAL INSTRUMENTATION (con't.)

Instrumentation Description	Manufacturer	Model	Serial Number	Installation Date
ENCOTEC I				
Metals Laboratory (con't.)				
ICP-Sequential	Perkin- Elmer	6500XL	129865	1987
ICP-Simultaneous	ARL	3560	5368	1989
ICP/MS Autosampler	Gilson	212	CK18459 SPO12095	1992
ICP/Mass Spectrometer	Perkin- Elmer	ELAN5000	0010	1992
General Chemistry and Inorganic Extractions Laboratories				
Analytical Balance	Mettler	AE240 - S	HO1673	1987
Analytical Balance	Mettler	PJ400	H1146	1985
Autoanalyzer	Lachat	QuikChemAE	1527	1992
Autotitrator	Schott Gerate	TR250	444519	1991
Block Digestor	Bran & Lubbe	BD40	G6017-9044	1990
COD Digestor	Hach	16400-10	8811111809	1989
COD Digestor	Hach	16500-10	3822	1981
Colorimetric Analyzer	Hach	DR/100	911200001983	1992
Colorimetric Analyzer	Hach	100	910453873	1991
Colorimetric Analyzer	Hach	100	860315965	1989
Colorimetric Autoanalyzer	Technicon	TRAACS 800	TC00218, TC0316	1988
Conductance Meter	YSI	32	90B012943	1990
Conductivity Meter	YSI	33	343	1982
Drying Oven	Baxter	DN-83	Not Available	1990
Drying Oven	Fisher	500	809-00-188	1989
Drying Oven	Thelco	28	21-AG-11	1980
Drying Oven	Thelco	28	21-AG-11	1980
Fourier Transform IR	Perkin-Elmer	1600	139205	1992
Oxygen Meter	YSI	57	484	1980
POX Analyzer	OIC	4420	601-6-331	1987
Rotary Extractor	Associated Design	3740-2-BRR	1057	1990
Rotary Extractor	Lars Lande	Not Available	Not Available	1988
Spectrophotometer	Hach	DR3000	890301869	1992
Steam Bath	Baxter	W2975-22	4901128	1991
Steam Bath	Precision Scientific	270	28AX1	1989

MAJOR ANALYTICAL INSTRUMENTATION (con't.)

Instrumentation Description	Manufacturer	Model	Serial Number	Installation Date
ENCOTEC I				
Gen. Chem. and Inorg. Ext. Labs (con't.)				
Steam Bath	Precision Scientific	Not Available	11AX-4	1987
TOC Analyzer	OIC	700	860-7-254	1987
Top Loading Balance	American Scientific	1200P	C0206266	1988
Turbidimeter	Hach	Ratio/XR	910602620	1992
pH Meter	Fisher	925	A032951	1989
pH/Ion Meter	Corning	250	2479	1984
Miscellaneous Support				
16' X 16' Walk-in Cooler	Kolpak	Not Available	Not Available	1990
20' X 14' Walk-in Cooler	Kolpak	Not Available	Not Available	1988
Deionization System (Super "Q")	Millipore	Not Available	ZD00022S4	1986
House Air Compressor System	Kaeser	SX - 6	120456	1991
Pancake GM Probe	Bicron	PGM	A480R	1990
Portable Survey Meter	Bicron	Surveyor 50	A409R	1990
Refrigeration System (2hp)	MIA	Not Available	610924L87	1990
Refrigeration System (2hp)	MIA	Not Available	610927L87	1988
Vacuum Pump	Alcatel	1030	150762	1990
Vacuum Pump	Alcatel	1030	36188	1990
Vacuum Pump	Alcatel	1030	45859	1990
Computer Software Support Equipment				
ARL Plasma Vision ICP Software	ARL	Version 2.0	Not Available	1989
Autotitrator TR600 Software	Schott-Gerate	Version 4.57	Not Available	1992
ENCOTEC Sample Management System Software	ENCOTEC	Version 6.0	Not Available	1989
Finnigan MAT Formaster II (CLP Deliverables) Organics Software	Finnigan MAT	Version 3.3	Not Available	1991
Finnigan MAT GC/MS SuperIncos Data System Software	Finnigan MAT	Version 9.0	Not Available	1989
Finnigan MAT GC/MS SuperIncos Data System Software	Finnigan MAT	Version 6.5	Not Available	1988
Fourier Transform IR T1600 Software	Perkin/Elmer	Data Spectra	Not Available	1992
Hewlett- Packard 5971A MSD GC/MS Software	Hewlett- Packard	Not Available	Not Available	1991
Ion Chromatography Software	Dionex	A1-450	Not Available	1992

MAJOR ANALYTICAL INSTRUMENTATION (con't.)

Instrumentation Description	Manufacturer	Model	Serial Number	Installation Date
ENCOTEC I				
Computer Software Support Equipment (con't.)				
Laboratory Information Management System	PE Nelson	Version 2.1.4	Not Available	1992
Lachat QuikChemAE Software	Lachat	Not Available	Not Available	1992
Nelson Analytical 2600 Batch Reprocessing Software	Nelson	Version 5.1	Not Available	1989
Nelson Analytical 2600 Chromatography Software	Nelson	Version 5.1	Not Available	1989
Perkin -Elmer ICP /6500 Software	Perkin-Elmer	Version 1.0	Not Available	1987
Perkin-Elmer 4100ZL Furnace Software	Perkin-Elmer	Version 6.2	Not Available	1991
Perkin-Elmer SCIEX ELAN5000 ICP-MS Software	JMI Software Cnslts	Version 2.0	Not Available	1992
Technicon TRAACS 8000 Ion Chromatography Software	Technicon	Version 4.1	Not Available	1986
Varian Saturn ITD - 40 Software	Varian	Not Available	Not Available	1991
Varian Spectra AA-20 Software	Varian	Issue 1	Not Available	1986
Ward Scientific (CLP Deliverables) Inorganics Forms Software	Ward Scientific	Version 2.65	Not Available	1990
ENCOTEC II (3965 Research Park Drive)				
GC/GC/MS Laboratory				
Automated Volatiles Concentrator, Purge and Trap	Tekmar	LCS - 2000	92078004	1992
Automated Volatiles Concentrator, Purge and Trap	Tekmar	ALS-2016	92073002	1992
GC Autosampler	Varian	8100	1214	1992
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Finnigan	Incos 500	IN0526	1990
Gas Chromatograph/ Mass Spectrometer, Quadrupole	Finnigan	4500	12366-0281	1992
Gas Chromatograph, Dual ECD	Varian	3400	15488	1992
Gas Chromatograph, Dual ECD	Varian	3400	15489	1992
Gas Chromatograph, ECD/FID	Varian	3700	11393-13	1992
GC Autosampler	Varian	8100	2529	1992
GC Autosampler	Varian	8100	2528	
GC Autosampler	Varian	8034	372224MY85	
Organic Extractions Laboratory				
Analytical Balance	Sartorius	BA310P	11203722	1992
Benchmate Workstation	Zymark	BFSI	4404	1992
Benchmate Workstation Collector	ISCO	Foxy 200	183234	1992

MAJOR ANALYTICAL INSTRUMENTATION (con't.)

Instrumentation Description	Manufacturer	Model	Serial Number	Installation Date
ENCOTEC II				
Organic Extractions Laboratory (con't.)				
Dessicator	Sanplatec	Drykeeper	N/A	1992
Drying Oven	Baxter	N-8620-13A	0292-0480	1992
GC Autosampler	Varian	8034	346508N084	1981
Gas Chromatograph, Dual ECD	Varian	3400	3006	1986
Gel Permeation Chromatograph	ABC Laboratories	1002	Not Available	1989
LC pump	SSI	300	E2904394	1992
Muffle Furnace	Thermolyne	F30420C	544920471676	1992
N-Evap. Evaporator	Organomation	III	11407	1992
Recorder	Yokogawa	29901	41PA0619	1992
Steam Bath	Boekel	1494	1077	1992
Tube Heater	Kontes	K720001-0000	1747	1992
UV Detector	Isco, Inc.	229	183121	1992
Ultrasonic Disrupter	Heat Systems	XL2020	G1810	1992
Vacuum Manifold	Supelco	12	N/A	1992
Water Recirculator	Brinkman/Lauda	UKT 1500 P	P14003	1992
Water Recirculator	Brinkman/Lauda	UKT 1500 P	P14002	
Water Recirculator	Brinkman/Lauda	MS3	N14006	
Water Recirculator	Brinkman/Lauda	MS3	N17049	
Water Recirculator	Brinkman/Lauda	MS3	P02027	
Water Recirculator	Brinkman/Lauda	MS3	N17050	
Water Recirculator	Brinkman/Lauda	MS3	P02024	
Water Recirculator	Brinkman/Lauda	MS3	N13019	
Water Recirculator	Brinkman/Lauda	MS3	N13020	
Water Recirculator	Brinkman/Lauda	MS3	N21007	
pH Meter	Corning	240	12632	
Waste Profile Laboratory				
Analytical Balance	Mettler	AJ100	M-73580	1992
Analytical Balance	Mettler	PM100	216326	1991
Analytical Balance	Denver Inst.	XE400	35802	1991

Instrumentation Description	Manufacturer	Model	Serial Number	Installation Date
ENCOTEC II				
Waste Profile Laboratory (con't.)				
Atomic Absorption Unit, Flame	Varian	AA20 ABQ	605 1195	1986
Ballistic Calorimeter	Gallenkamp	CBB330030	581190	1991
Box Furnace	Lindberg	51-849	84-54142	1991
Drying Oven	VWR	1350ED	1004694	1991
Isoperibol Calorimeter	Parr	1261EA	788	1991
Pensky-Martens Flashpoint Analyzer	UIC	HER200	90892	1991
Pensky-Martens Flashpoint Analyzer	UIC	HER200	89619	1989
Portable Scintillation Counter	Lludlum	44-2	PR079158	1991
Portable Survey Meter	Lludlum	3	84799	1991
Rotary Extractor (TCLP)	Lars Linde	N/A	1318	1992
Rotary Extractor (ZHE)	AGS Design	3740-8-BRE	1309	1992
Rotational Viscometer	Brookfield	LVTDV-I	821248	1991
Soil Carbon Analyzer	Dohrman	PIR2000	Not Available	1989
Soil TOC Furnace	Dohrman	183-SS	HI3506	1989
Top Loading Balance	Mettler	P1400	H11456	1990
Top Loading Balance	Sartorius	B610OUR	39029023	1989
Ultrasonic Cleaner	Branson	B1200 R-4	F0695211442	1992
Water Bath	Lab-Line	W2975-22	0490-1128	1990
pH Meter	Orion	250A	2058	1991
Miscellaneous Support				
Refrigerators	Kenmore	363.9601416	various	1992
Vacuum Pump	Alcatel	1101101416	79324	
Deionization System (Super "Q")	Millipore	LD0F01205	1106170402	1992

Appendix B

The following are provided as examples of forms incorporated into the production of each set of analytical data. These forms are derived from those satisfying the EPA Contract Laboratory Program's requirement for data packages in very specific order and format. The organization sheets provide structure to the data package submitted for review, ensuring that all data is presented in a specified sequence and that all necessary Quality Control analyses and checks have been performed. In addition to ensuring consistency in the documentation from one data set to the next, these forms provide the reviewer with a data package which is more readily assessed for data quality and completeness.

DATA ORGANIZATION SHEET

DATA PACKAGE: PRIVATE CLIENT

1) Folder tab with: Client name, project number, number of samples, Parameter and Matrix ☐

2) Package Contents: (in chronological order)

- A) Project sheet(s) ☐
- B) Extraction information sheet(s) (BNAs) ☐
- C) Dilution logs ☐
- D) Surrogate recovery form(s) ☐
- E) Matrix Spike recovery form(s) ☐
- F) Lab Control Spike recovery form(s) ☐
- G) BFB or DFTPP form(s) ☐
- H) Standards calibration sheet(s) ☐
- I) Standards ☐
- J) Blanks ☐
- K) Samples (sequential by ENCOTEC #)
  - 1) "Best" dilution of sample first followed by other dilutions in chronological order ☐
  - 2) Individual sample package (stapled) ☐
    - a) Quan report (with any dilutions and peak ht of TICs and IS written on report)
    - b) Chromatogram (RIC) with IS, SUR, TICs and analyzed compounds labeled ☐
    - c) Library searches ☐

DATA REVIEW

	INITIALS
SECONDARY REVIEW	☐
TERTIARY REVIEW	☐

DATA SIGN OFF

THE DATA PACKAGE HAS BEEN ORGANIZED AS STATED ABOVE.

ANALYST _____ DATE _____

ENCOTEC

ENCOTEC

DATA ORGANIZATION SHEET - EPA/CLP CASE SIGN OFF

1) Folder Tab with : 1) Client Name; 2) Project No.; 3) Case No.
4) # of Samples ; 5) Parameter; 6) Matrix ☐

2) Project Sheet ☐

3) Extraction Logs ☐

4) Case Narrative ☐

5) Surrogate Percent Recovery Form ☐

6) Matrix Spike Recovery Form ☐

7) Package Contents (in order by Quan.91 files)

- A) Daily Review Checklist ☐
- B) Dilution Log ☐
- C) CLP Run Log ☐
- D) Standard Calibration Sheet ☐
- E) DFTPP Form ☐
- F) Diagnostic Report of High Level Std. ☐
- G) Standards ☐
- H) Blanks ☐
- I) Samples : ☐
  - I.1) Chromatogram -- labeled ☐
  - I.2) Quan report -- with dilution & peak height of TIC's and IS written on report ☐
  - I.3) Labeled Dual Spec. & Spec. for each compound in elution order ☐
  - I.4) Library searches in elution order ☐

List *.91 files that correspond to this case below :

_____	_____
_____	_____
_____	_____
_____	_____

This Data Package has been organized as stated above

Analyst: _____ Date: _____

This Package has been reviewed and is complete.

Senior Chemist: _____ Date: _____

Tertiary Reviewer: _____ Date: _____

signoff.xls/dt

ENCOTEC

DATA ORGANIZATION SHEET - EPA/CLP CASE SIGN OFF

1) Folder Tab with : 1) Client Name; 2) Project No.; 3) Case No.
4) # of Samples ; 5) Parameter; 6) Matrix ☐

2) Project Sheet ☐

3) Extraction Logs ☐

4) Case Narrative ☐

5) Surrogate Percent Recovery Form ☐

6) Matrix Spike Recovery Form ☐

7) Package Contents (in order by Quan.91 files)

- A) Daily Review Checklist ☐
- B) Dilution Log ☐
- C) CLP Run Log ☐
- D) Standard Calibration Sheet ☐
- E) DFTPP Form ☐
- F) Diagnostic Report of High Level Std. ☐
- G) Standards ☐
- H) Blanks ☐
- I) Samples :
  - I.1) Chromatogram -- labeled ☐
  - I.2) Quan report -- with dilution & peak height of TIC's and IS written on report ☐
  - I.3) Labeled Dual Spec. & Spec. for each compound in elution order ☐
  - I.4) Library searches in elution order ☐

List *.91 files that correspond to this case below :

_____	_____
_____	_____
_____	_____
_____	_____

This Data Package has been organized as stated above

Analyst: _____ Date: _____

This Package has been reviewed and is complete.

Senior Chemist: _____ Date: _____

Tertiary Reviewer: _____ Date: _____

signoff.xls/dt

GC/HPLC Laboratory: Data Packet Organization Form

Data Packet ID: _____ Analysis Type: _____

Please organize data packet as follows:

I DATA FORMS: (Each Submission ID paper-clip together)

1) Data sheet(s) (sequential by Encotec numbers) _____
2) Method Blank(s) _____
3) Copy of LCS(s), LCS/LCD(s)
4) Copy of MS/MSD(s) _____
5) Project Sheet (with run # in upper right hand corner) _____

II ANALYSIS COMPLIANCE: (Paper-clip together)

1) All Analysis Compliance and Corrective Action forms _____
2) EXPANDED Sequence Table(s) _____
3) All method print-outs with component tables and calibration reports. Labeled Quant. or Conf. _____
4) Other Required Compliance Forms:

- Continuing Calibration Forms _____
- Herbicide Derivitization _____
- Pesticide Breakdown _____
- Injection Summary Form _____
- Extraction Forms _____

5) Any other forms: _____ _____

III QC FORMS: (Paper-clip together)

1) Copy of all surrogate recovery forms submitted with data sheets _____
2) Original LCS, LCD, MS, MSD forms for review and archival _____

IV CHROMATOGRAMS:

1) In sequential order with quantitation channel first _____
2) All calculations for positives, with units, on C-gram _____
3) All compounds IDed on External Standard Reports flagged with either a "+", "-", ">25%" or "<DL" _____

This data packet is complete and in compliance with analytical protocol. Any deviation or exceptions to protocol have been authorized and are noted on the ANALYSIS COMPLIANCE and/or CORRECTIVE ACTION forms included in this data packet.

Analyzed by: _____ Date: ____/____/____

Reviewed by: _____ Date: ____/____/____

Environmental Control Technology Corporation

INORGANICS DEPARTMENT

Raw Data - Cover Sheet

Date of Analysis: __/__/__

Parameter

Analyst: _____

Clients included in this data package

	Client	Job #		Client	Job #
1.			4.		
2.			5.		
3.			6.		

	TIER I REVIEW - (Analyst)	Comments:
	☐ Calculations are accurate & complete	
__/__/__	☐ QC frequency is satisfied	
	☐ QC group requirements are satisfied	
	☐ All appropriate instrument printouts are attached	
	☐ Analysis has been scrubbed "A --->D"	

	TIER II REVIEW - (Senior Analyst)	Comments:
	☐ ICV/CCV; ICB/CCB; PB/LCS checked	
	☐ 10% Calculation review completed	
__/__/__	☐ Quality Control frequency checked	
	☐ All rejected results have been documented	
	☐ See comments for future reference	

	TIER III REVIEW - (Data Management)	Comments:
	☐ 20% Calculation review completed	
	☐ Quality Control review completed	
	☐ Data accepted	
	☐ Data rejected	
__/__/__	☐ All rejected results have been documented and rescheduled	
	☐ Data is summarized	
	☐ See comments for future reference	

INORGANICS GROUP
DATA MANAGEMENT ROUTING SHEET

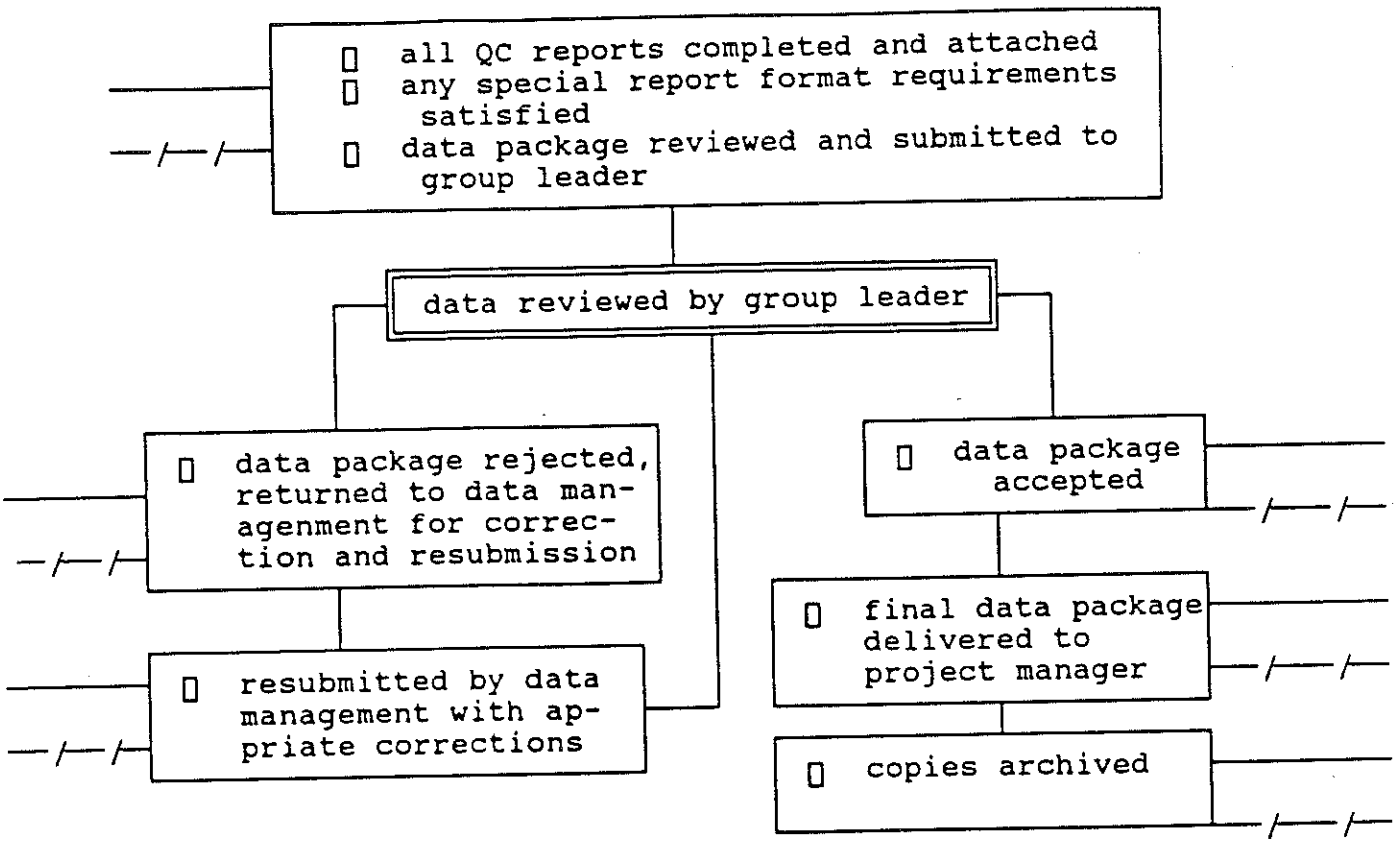
BATCH:_____ DATE RECEIVED:___/___/____

CLIENT:_____

GENERAL CHEMISTRY ☐ METALS ☐

QC REPORT NEEDED? ☐ YES ☐ NO

QC GROUP ASSIGNMENT (if any):_____

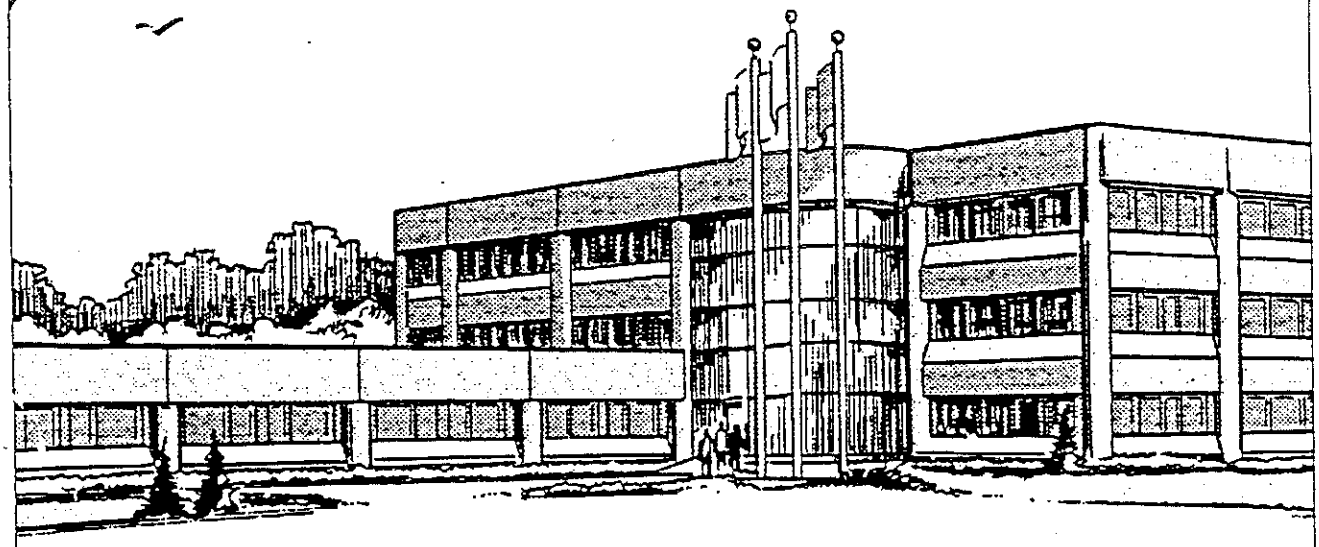


DATE	EVENT	TO WHOM	DATE	EVENT	TO WHOM

RTNGSLP.frm 07/12/90

WW ENGINEERING & SCIENCE

Environmental Laboratory Division



Quality Assurance / Quality Control Procedures Manual

WW Engineering & Science

WW ENGINEERING & SCIENCE ENVIRONMENTAL LABORATORY DIVISION
QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES MANUAL

TABLE OF CONTENTS

1.0 PURPOSE OF THE MANUAL

1.0 PURPOSE OF THE MANUAL

The purpose of this manual is to specify procedures and technical requirements to be used by the WW Engineering & Science (WWES) environmental laboratories to assure that the data generated by the laboratory is accurate, reproducible and timely. This manual provides the chemistry laboratory a quality control plan which is to be used by every individual involved in the analytical efforts at WWES.

1.1 THE NEED FOR ANALYTICAL QUALITY CONTROL

There is a growing importance attached to the measurement of the concentration of any contaminant in water, effluents, and solid samples. As with any type of measurement the results of the methods utilized to measure the concentrations of these contaminants generally differ from the true concentration, i.e. all results are subject to error. Many experimental studies have shown that errors can arise which are as large as a 50 percent variations from the true value, and in fact, may vary between laboratories. Inaccurate analytical results restrict the ability of the analyst and the recipient of the data to draw valid conclusions and usually lead to false or misleading conclusions. Examples of common problems which arise during an analytical effort are as follows:

A. Results, which are compared between two or more laboratories, are in error relative to each other.

B. Results are to be used to decide if a water quality standard has been observed especially as the level of the analysis approaches the detection level.

C. An inappropriate test procedure has been used to determine the analyte, resulting in values that do not represent the true sample concentration, i.e. direct aspiration of a turbid sample.

There is also increasing concern about the control of these errors being expressed at the local, the national and the international levels. The concern centers around the need to have a maximum amount of valid information obtained in a cost effective manner. In order to control errors, it is necessary to be able to measure the magnitude of these errors. This manual identifies the activities that are involved in the measurement and control of error. WWES considers analytical quality control of great importance, and requires that it be a primary feature in any analytical effort. The WWES requirements for analytical quality control are in concert with the quality control needs and demands of other organizations, such as the Environmental Protection Agency, various state regulatory agencies, and private industry.

Approximately twenty to thirty percent of all the available effort for routine analysis is absorbed in the execution of quality control requirements. It is often argued that the extent of this effort is too great with respect to routine laboratories and their need to be

profitable in their operation. The argument therefore claims that extensive quality control is an impractical expectation for a routine laboratory. However, the corporate policy at WWES demands that the appropriate level of quality control be applied to all analytical effort at WWES, regardless of the sample lot.

In the total effort, it is preferable to obtain twenty to thirty percent fewer results of known accuracy for each analytical batch than it is to obtain larger numbers of results of undefined accuracy. Due to the fact that all analytical procedures are subject to errors derived from many sources, it is not reasonable to assume that quality control is unnecessary with a "good" analyst. However, even a "good" analyst may not have an adequate idea of his (her) accuracy. Multiple studies by the EPA, both within laboratories and between laboratories, has shown this reasoning to be generally unsound.

2.0 QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

2.0 QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

2.1 OBJECTIVE OF THE QA PROGRAM AT WWES

The purpose of the quality control program is to continuously monitor error, both random and systematic, which inhibits the production of reliable and defensive analytical data. Error is inherent in any analytical routine, even with the most rigorous controls, and thus, a good QC program addresses not only the basic control techniques but also statistical means of measuring precision and accuracy and the confidence limits on these measurements.

The purpose of this manual is to specify the procedures, records, Chain-of-Command, and technical requirements which will be adhered to by the laboratories of WWES.

2.2 ORGANIZATION

Quality control at WWES begins with the bench analyst and moves up through the Chain of Command ultimately residing at the level of the President. A QC program which is administered only at the upper levels of management is doomed to failure and is unfair to the bench level analyst who needs a means by which he can observe the quality of his work. Quality Control is a two way program at WWES where directives from management are as important as suggestions and assistance from the bench analyst.

2.2.1 QC Chain of Command Flow Chart

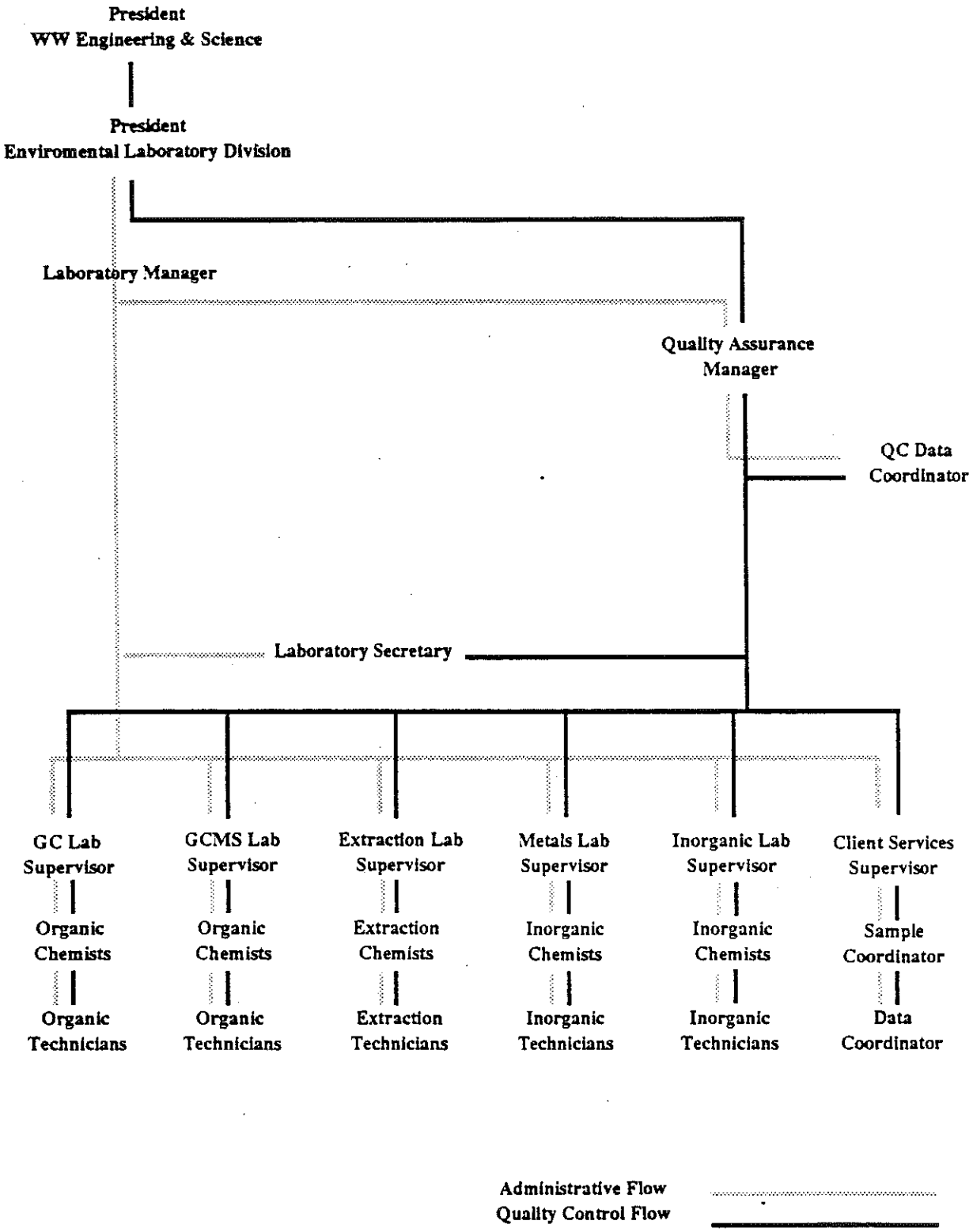
The following flow chart represents both the QA Chain of Command (solid line) and the Administrative Chain of Command (dotted). The flow chart represents the philosophy of WWES relative to the interaction of QC and production. Although the QC Manager reports to the Director of Analytical Services in the supervisory Chain-of-Command, his responsibilities for quality control require that he answer to the President of the Environmental Laboratory Division. The QC Manager acts as an immediate record keeper, QC administrator and liaison to the lab manager.

However, when a question relative to the quality of analytical data arises, the QA Manager, in conjunction with the President of the Environmental Laboratory Division, has the right to prevent data dissemination. In cases of conflict, the President of the Environmental Laboratory Division has final authority except when a compromise or directive is issued by the President of WW Engineering & Science.

2.2.2 Responsibilities of the Laboratory QC Manager

2.2.2.1 To monitor the Quality Assurance activities in the laboratory insuring adherence to all policies and procedures.

QUALITY CONTROL CHAIN OF COMMAND FLOW CHART



2.2.2.2 To identify problem areas and help in recommending improvement and changes.

2.2.2.3 To keep abreast of changing development in analytical QC particularly requirements set by regulatory agencies.

2.2.2.4 To arrange or produce random blind control samples.

2.2.2.5 To approve all laboratory data prior to recording such data for report generation purposes.

2.2.2.6 To maintain QC on all analytical activities and update control limits in a timely manner.

2.2.2.7 To oversee the maintenance of balance and controlled temperature apparatus record books on a daily basis and insure that such records are maintained on every piece of equipment.

2.2.2.8 To assure that bottle preparation, approval and storage meet established criteria.

2.2.3 Responsibilities of the Sample Coordinator

A full position description of the Sample Coordinator can be found in the "WWES Sample Receiving SOP".

- To insure that all samples received at WWES are properly preserved, split, logged-in, and stored in agreement with the log-in manual.
- To insure that all COC shipments are handled according to established procedures including storage, sample tracking and completion of files.
- To insure that all project sheets and subsequent paperwork is completed and filed.
- To insure that labile samples are distributed in a timely manner.
- To cooperate with the QC Manager in introducing blind samples.

2.2.4 Responsibilities of the Analytical Staff

- To insure that all records are generated and recorded on a daily basis.
- To insure that the following bench level QC requirements are met. To fill out lab notebooks daily as required. To provide for QC on every batch of samples. This level of effort generally includes:
  1) An initial calibration blanks and standards
  2) 10% sample matrix spikes

3) 10% sample matrix duplicates
4) Laboratory control sample and a method preparation blank for each batch.

- To insure that instruments are calibrated prior to initiating any analysis and that no analyses are started unless calibration has been satisfactorily completed.
- To insure that every batch of analyses meets established QC guidelines or is reanalyzed automatically.
- To inform the lab manager of any reoccurring problems or systematic trends which may effect quality.

2.2.5 Responsibilities of the Laboratory Supervisor

- To insure that sufficient competent staff is available to administer QC.
- To insure that all participating analysts are certified in the test they are performing.
- To insure that effective training and orientation takes place for every new analyst.
- To insure that all QC procedures, directives or project oriented requirements are met.
- To review all preliminary reports and approve them prior to the generation of a final report.
- To interface with the QC coordinator and QC Manager on a routine and consistent basis.
- To take responsibility for immediate solutions to QC problems which may slow or stop production.

2.2.6 Responsibilities of the Data Coordinator

The Data Coordinator's (DC) responsibilities are:

- To enter all data generated into the appropriate records.
- To insure that the Laboratory Supervisor has signed the data forms (bench sheets prior to entry).
- To inform the QC Manager when a project is complete and ready for a preliminary report.
- To provide corrections to all reports from preliminary report feedback.

3.0 FACILITIES AND EQUIPMENT

3.0 FACILITIES AND EQUIPMENT

3.1 The physical plant layout diagram is enclosed. The approximate square footage allocated to each analysis area is presented as well as the number of personnel normally working in each area. A listing of equipment presently utilized by WWES is also enclosed.

3.2 The quality of the analytical instrumentation utilized by WWES is of great importance considering its ultimate effect on data quality. The following guidelines exist for the procurement of analytical instrumentation:

3.2.1 Equipment Need

An equipment need is identified by the Lab Manager or the President as a result of:

- o New Contractural Effort
- o Regulatory Changes
- o Normal Upgrade/Replacement
- o Capacity Improvements

3.2.2 Procurement Procedure

The performance specifications defined by the need are used to identify prospective equipment suppliers. The Lab Manager mails the performance specifications to the prospective equipment suppliers. Those suppliers able to meet the performance specifications are asked to provide a quotation for the purchase or lease of the equipment. An evaluation of the quotations is made by the Lab Manager with consideration given to such items as: equipment ease of use, degree of automation, specification compliance, potential for computerization, price and space requirements.

A written recommendation by the Lab Manager is presented to the President for their review and comment.

A final recommendation by the President of the Laboratory Division is made to the Vice President of Corporate Finance. The final approval is granted based on the assurance of complying with all regulatory and corporate guidelines for the generation of the highest quality data.

3.3 CHEMICAL PROCUREMENT AND INVENTORY PROCEDURE

All reagent specifications are dictated by the EPA/APHA or NIOSH approve analytical methods. These reagent specifications are identified and maintained on the chemical inventory index card system. The chemical inventory system assures the order of chemical use and minimizes the possibility of exceeding their useful shelf life. The

addition of a new method or a change in an existing method that requires a corresponding addition or change in a reagent used for that method will be identified by the Lab Area Supervisor or Group Leader. The Supervisor or Group Leader will update the chemical inventory.

All reagent specifications including available vendors are listed in the chemical inventory tables.

All chemical reagents are received by a representative from the appropriate lab area. The representative opens the shipping package and compares the packing slip with the contents. Discrepancies are identified to the Area Supervisor. The materials receipt is identified and recorded on the chemical inventory. The materials are then inventoried on the chemical inventory index. The index system identifies the amount(s) received and when. When the last bottle/container of the chemical remains, the chemical is placed on an open order sheet located in the lab.

The group leader/supervisor for that lab area is responsible for picking up the chemical open order sheet each week and preparing a purchase order. The purchase order is approved by the Lab Manager and a typed purchase order is issued to the approved vendor that has been previously identified as being able to supply the specified material. The receipt of the new order initiates the inventory system activities.

3.4 PREVENTATIVE MAINTENANCE

Every analytical instrument has a separate maintenance log book as identified in the Document Control Section No. 7.3.9.. The required maintenance activities have been developed by the Lab Manager and each Group Leader/Area Supervisor. The maintenance activities comply with manufacturer specifications and working experience requirements.

Each maintenance log book contains a table indicating the frequency and type of maintenance required. The maintenance activity is documented each day or as the frequency requirements dictate.

Analysts are assigned the responsibility of maintaining various instruments or equipment in their respective laboratory areas. The Group Leader or Area Supervisor is responsible for checking the maintenance log books each week and signing off as checked. The Quality Assurance Manager is notified of any deviations or lack of maintenance activity performance, and corrective actions are taken.

ANALYTICAL EQUIPMENT

WWES-INORGANIC LABORATORY

Instrument Description	Manufacturer	Model No.
Analytical Balance (2)	Metler	AE163
Analytical Balance	Metler	PC4400
Analytical Balance	Metler	BB2400
Auto-Analyzer (dual channel)	Bran & Lubbe	TRAACS 800
Auto-Analyzer	Technicon	AAII
Auto-Analyzer	Lachat	Quick Chem
Conductivity Meter	YSI	32
FTIR Spectrophotometer	Perkin-Elmer	1600
pH/mv Meter	Beckman/Altrex	70
pH/mw/ISE Meter	Orion	EA920
Spectrophotometer (visible)	Hitachi	100-40
Spectrophotometer (UV-VIS)	Shimadzu	1604
Spectrophotometer (UV-VIS)	Shimadzu	UV-1201
Total Organic Carbon Analyzer (TOC)	O.I.C.	700
Total Organic Halogen Analyzer (TOX)	Xertex/Dohrman	---
Nephlometer	HACH	2100A
Polarograph	EG&G Princeton Applied Research	384B
Auto-Titrator	Metler	DL12

( ) Designates multiple units.

ANALYTICAL EQUIPMENT

WWES-METALS LABORATORY

Instrument Description	Manufacturer	Model No.
AA Spectrophotomer (flame)	Perkin Elmer	5000
AA Spectrophotomer (flame/furnace)	Perkin Elmer	5100 PC
AA Spectrophotometer (furnace)	Perkin Elmer	5100 PC
ICP Spectrophotometer	Perkin Elmer	Plasma 40
ICP Spectrophotometer	Perkin Elmer	Plasma 400
Autosampler	Perkin Elmer	AS-50
Autosampler	Perkin Elmer	AS-51
Autosampler	Perkin Elmer	AS-60
Autosampler	Perkin Elmer	FIAS-200
Mercury Analgam System	Perkin Elmer	---
Microwave Digestion System	CEM	MDS 810
Ultrasonic Nebulizer	CTech	---

ANALYTICAL EQUIPMENT

WWES-GC LABORATORY

Instrument Description	Manufacturer	Model No.
Gas Chromatographs (3) w/ECD (2), PID/FID, FID/ECD	Varian	3700
Gas Chromatograph (3) w/FID/CED	Varian	3400
Gas Chromatograph (2) w/Hall-PID	Tracor	540
Gas Chromatograph (2) w/Hall-PID	Tracor	585
Gas Chromatograph w/Hall-PID	Tracor	9000
Gas Chromatograph w/FID	HNU	301
Autosampler	Varian	8000
Autosampler (3) w/Auto Sample Heater	Tekmar	ALS 2016
Concentrator (4)	Tekmar	LCS 2000
Autosampler	Tekmar	ALS 2050

( ) Designates multiple units.

ANALYTICAL EQUIPMENT
WWES-GC LABORATORY

Instrument Description	Manufacturer	Model No.
Autosampler (3)	Tekmar	ALS (10 place)
Concentrator (3)	Tekmar	LSC-2
Thermal Tube Desorber	Envirochem, Inc.	850
HPLC (UV)	Isco	2300
HPLC (UV-Fluorescence)	Perkin Elmer	Series 410
LC Oven	Perkin Elmer	101
Diode Acray Detector	Perkin Elmer	235
Fluorescence Detector	Perkin Elmer	LC240

Chromatography Data Systems:

Integrators (7)	Spectra Physics
Turbochrom	Perkin Elmer

( ) Designates multiple units.

ANALYTICAL EQUIPMENT

WWES-GC/MS LABORATORY

Instrument Description	Manufacturer	Model No.
Mass Spectrometer (2)	Finnigan Mat	OWA 1000
Mass Spectrometer (2)	Extrel	ELQ-400
Mass Spectrometer (2)	Varian	Saturn
Autosampler (2)	Varian	8000
Gas Chromatograph (2)	Varian	3400
Gas Chromatograph (2)	Perkin Elmer	Sigma 3B
Gas Chromatograph w/FID	Varian	3400
Autosampler	Tekmar	ALS 2016
Concentrator	Tekmar	LCS 2000
Concentrator	Tekmar	LCS-2
Concentrator	Tekmar	LCS-2000

() Designates Multiple Units

COMPUTING EQUIPMENT

WWES-ALL LABORATORY AREAS

Instrument Description	Manufacturer	Model No.
Mini-Computer	Digital Equipment	Micro-Vax II
Personal Computer (18)	Club	386
Personal Computer (7)	Club	286
Personal Computer (3)	Everex Step	386
Personal Computer (2)	Macintosh	Plus
Personal Computer	Macintosh	SE
Personal Computer (2)	Compac	386
Personal Computer (2)	Zenth	386
Personal Computer	Epson	Equity III
Printer (3)	Pentronix	6280
Printer (2)	Hewlet Packard	Laser Jet
Printer	Hewlet Packard	Laser Jet II
Printer	Hewlet Packard	Laser Jet IIID
Printer (6)	Epson	FX-850
Printer	NEC-Laser Silent Writer	LC890

( ) Designates multiple units.

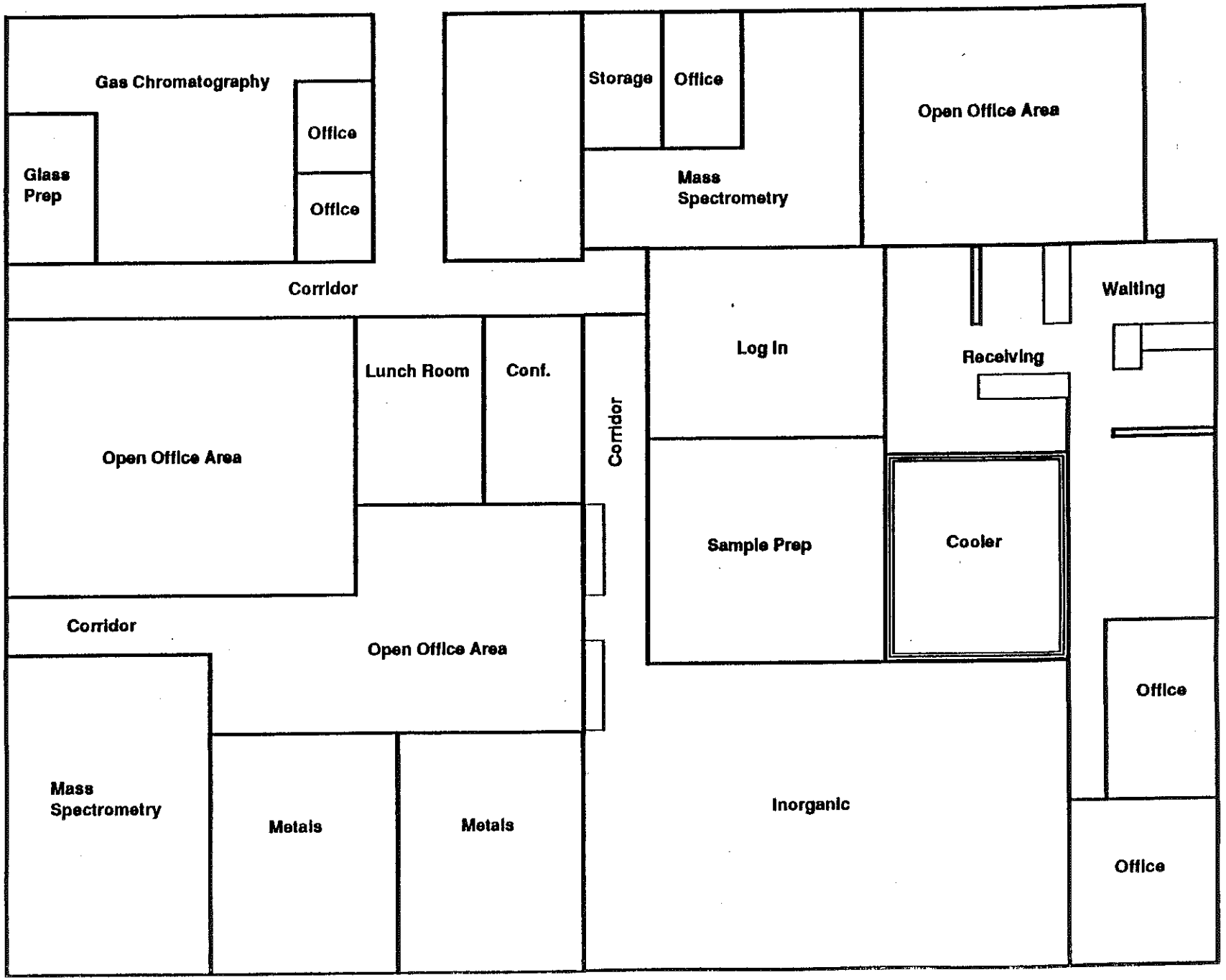
PHYSICAL PLANT:

Laboratory Name:	Environmental Laboratory Division WW Engineering & Science, Inc.
Address	5555 Glenwood Hills Parkway S.E. Grand Rapids, MI 49588
President	Richard R. Rediske, Ph.D.
Vice President/Lab Manager	John P. Dullaghan, MBA

An attached drawing of the laboratory indicates the general areas of analysis, the space allotted to each, and the number of personnel generally assigned to each area.

Analysis	Space Allotted, Ft^2	No. of Personnel (September, 1991)
Wet Chemistry/Microbiology	Approx. 2000	12
Atomic Absorption/Emission	Approx. 2000	12
Gas Chromatography (GC)	Approx. 2400	10
GC/Mass Spectrometry	Approx. 1500	8
Sample Processing & Storage	Approx. 1500	5
Administrative Offices	Approx. 2500	20
Organic Pretreatment	Approx. 1000	6
Laboratory Offices	Approx. 1000	10

WW Engineering & Science Environmental Laboratory Division



4.0 ANALYTICAL METHODOLOGIES

4.0 ANALYTICAL METHODOLOGIES

4.1 METHODS UTILIZED

The WWES Laboratory maintains and updated reference volumes of approved analytical methodologies for environmental and non-environmental analysis. A responsibility of the Lab Manager and the President of the Environmental Laboratory is to continually seek and review regulatory method changes and their impact on current laboratory practices. The most commonly referenced materials include:

- "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020 revised March, 1983.
- "Manual of Analytical Procedures" NIOSH, Volumes 1 & 2, Third Edition, Feb., 1984.
- Standard Methods for the Evaluation of Water and Wastewater 17th Edition, APHA, AWWA, WPCF; 1989..
- "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA 600/4-79-019, March 1979.
- "Physical and Chemical Methods for the Evaluation of Solid Waste" EPA-SW846 Third Edition, 1990.
- "Guidelines Establishing Text Procedures for the Analysis of Pollutants". CFR July 1, 1990.

4.2 METHOD CALIBRATION AND OPERATING PROCEDURES

A standard operating procedure manual exists for all analytical procedures. The S.O.P's include specific calibration procedures that must be followed by an analyst prior to conducting sample analysis. The analyst is required to perform and document the calibration procedure. The calibration activity is identified by each analyst in their lab notebooks. The actual standards utilized are found in each instrument log book. It is the responsibility of each analyst to document all calibration and operating procedures utilized in the instrument log books. It is the responsibility of the group leaders and/or supervisors to notify the Quality Assurance Manager when deviations occur so that corrective actions can be taken. The corrective action will be to identify whether the information is simply missing (not entered) and to have it recorded or if the calibration has not been performed, to not release data generated that day and require those samples to be rerun.

5.0 METHOD CERTIFICATION

5.0 METHOD CERTIFICATION

All methods used by WWES which were not developed by WWES will be certified prior to their use. Method Certification is contiguous with the certification of the analyst and requires essentially the same analytical program. Method certification is necessary in order to establish detection limits, method application limits and criteria for control limits. In most cases, detection limits and recoveries stated in a method are obtained under ideal conditions and do not reflect real world solutions, i.e.. silty well water and industrial effluent versus a drinking water supply. Method certification falls into 2 categories: 1) Methods being employed for the first time and 2) Methods which are to replace currently certified methods (replacement methods). In either case, analysis of client sample may not proceed until certification has occurred.

5.1 METHOD CERTIFICATION

5.1.1 Linear Range

The first step in certifying a method is to establish the linear range (operating range) of the method. A method may be used only over the range in which it is linear. Some methods do not have linear ranges but curves from which results are calculated. For the moment we will ignore methods with curves. A linear range must be established independent of the method data since instruments can effect the range. Standards and multiple detections will be used for establishing the linear range. For example, a range of 1 to 1000 has 3 decades (3 orders of magnitude or 103). Therefore, a range of 1 to 1000 requires 11 levels of test standards (.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, 1000). Notice that each decade follows the 0.5x to 10x rule, i.e. the area 10 to 100 is covered by 5,10,20,50 and 100. The range to be attempted is dependent on the method, the instrument and the analytical supervisor. If the responses show linearity, the range has been established. If, however, a curve develops or there appear to be two linear ranges, the standards must be repeated including additional levels to verify the status of the questionable area.

5.1.2 Working Curves

Some methods operate from a curve response, i.e. sodium by emission spectroscopy. The method will indicate the working curve which must be verified. The method with working curves requires a full curve each time an analysis is to be performed.

5.1.3 The Generation of the Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that

the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

SCOPE AND APPLICATION

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete , specific and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device or instrument independent.

PROCEDURE

1. Make an estimate of the detection limit using one of the following:

   (a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. if the criteria for qualitative identification of the analyte is based upon pattern recognition techniques, the least abundant signal necessary to achieve identification must be considered in making the estimate (PCB).

   (b) The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.

   (c) The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations i.e. a break in the slope of the standard curve.

   (d) The concentration value that corresponds to known instrumental limitations.

   It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent water (blank) prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL (Recommend between 1 and 5 times the estimated MDL) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL proceed to Step 4.

If the measured level of analyte is greater than five times the estimated MDL, add a known amount of analyte to bring the concentration of analyte to between one and five times the MDL in the case where an interference is co-analyzed with the analyte.

If the measured level of analyte is greater than five times the estimated MDL there are two options:

(1) Obtain another sample of lower level of analyte in same matrix if possible.

(2) The sample may be used as is for determining the MDL if the analyte level does not exceed 20 times the MDL of the analyte in reagent water. The variance of the analytical method changes as in the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If blank measurements are required to calculate the measured level of

analyte, obtain separate blank measurements for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 4a. This will: (1) prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less than five times the calculated MDL. To insure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:

(1) If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.

(2) If these measurements indicate the sample is not in the correct range, re-estimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance (S^2) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i\right)^2 / n\right]$$

where the x1i = 1 to n are the analytical results in the final method reporting units obtained from the n sample aliquots and X,2 refers to the sum of the X values from i = 1 to n.

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1),-a=.99)} \cdot S$$

where:

MDL - the method detection

$t_{(n-1,1-a=.99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence limits for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (X2/df) and calculated as follows:

MDLlcl = 0.64 MDL
MDLucl = 2.20 MDL

where MDLlcl and MDLucl are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimated MDL in Step 1, take the MDL as calculated in Step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with Step 4.

(b) If the current MDL determination is an iteration of the MDL procedure for which the spiking level does not permit qualitative identification, report the MDL as that concentration between the current spike level and the previous spike level which allows qualitative identification.

(c) If the current MDL determination is an iteration of the MDL procedure and the spiking level allows qualitative identification, use S2 from the current MDL calculation and S2 from the previous MDL calculation to compute the F ratio.

if $S^2_A/S^2_B < 3.05$

then compute the pooled standard deviation by the following equation:

$$\text{Spooled} = \left[\frac{6S^2_A + 6S^2_B}{12} \right]^{1/2}$$

if $S^2_A/S^2_B > 3.05$, respike at the last calculated MDL and process the samples through the procedure starting with step 4.

(c) Use the Spooled as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681 \text{ (Spooled)}$$

where 2.681 is equal to t(12,1-a = .99)

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

$$MDLlcl = 0.72 \text{ MDL}$$
$$MDLucl = 1.65 \text{ MDL}$$

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

REPORTING

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units, if the analytical method permits options which affect the method detection limit these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with the MDL value. Report the mean analyte level with the MDL if a laboratory standard or a sample that contained a known amount analyte was used for this determination, report the mean recovery, and indicate if the MDL determination was iterated.

If the level of the analyte in the sample matrix exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL

REFERENCE

40 CFR Part 136 Appendix B, USEPA Chapter 1, 7/1/90.

Table of Students t Values at the 99 Percent Confidence Level

Number of Replicates	Degrees of Freedom (n-1)	$t_{(n-1,1-oc=.99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
		2.326

5.1.4 Method Spikes

Method spikes will be carried out over 2 separate days at the specified levels including blanks and calibration standards. The data obtained at the 2x or 5x level (for each certified range) will be used to establish a mean and standard deviation for initial control charts. Once this data has been generated and approved by the analytical manager, the method has preliminary certification and is ready for application to real world samples. These control limits will be updated with every batch of samples until 30 numbers have been developed to establish reliable control limits. After 30 data points the DC will provide updated control limits with each additional 20 numbers.

5.2. REPLACEMENT METHOD CERTIFICATION

When a new method is to be employed (where a new method is defined as including a new instrument method, i.e. flame vs flameless AA or Hall vs ECD), the method must be certified prior to its use on client samples.

Certification follows the procedures described in Sections 5.1. The results of these tests are important but are not necessarily compared to the current method. The detection limit may change and the work range may change but if they meet the needs of the lab, these changes are to be ignored. One may elect to utilize a t-test analysis to identify the method differences as being significant or not.

5.2.1 Comparison by the t-Test

One sample will be analyzed a minimum of 4 times by each method. The results will be subject to a t-test analyses. If the t-test indicates statistical correlation, regardless of the correlation coefficient, the new method is certified. If the t-test fails, refer to Section 5.2.2 below.

5.2.2 Decisions on Certification

The purpose of a new method is to improve accuracy, precision and efficiency. Efficiency is of no consequence if a method is imprecise and inaccurate, and therefore, is not a consideration in certifying new methods. However, a new method may fail the t-test because it is more accurate and/or more precise. Careful consideration and more analyses may be necessary with a new method by the analytical manager and supervisor.